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-----claim tree-----

1----2----4----5
+----19
+----18
+----15----17
+----14
+----13
+----12
+----11----16
+----10
+----9
+----8
+----7
+----6
+----3

20

21

22

-----112-----

-----best-----

6323169;6492311;6524376;6264839;4105783
6410494
4197316
5994290
4338204
4238192
4292035
6365564
4642197
6479448
5521293
5710121
6423676
6514925
4689168
6399557
6387862
4371517
6093343
6369024
6395701
6239093
6228829
4597898
6087309
6376450
6046152
6513433
6117357
6525041
6306812
6387927
4102823
6218351
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6514926
6514944
6103678
6510877
4493773
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5565145
6495129
6495357
5245072

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4870038

-----classlist-----

510/175

510/176

134/3

510/432

510/421

134/2

510/475

510/245

510/177

134/41

134/40

510/372

510/499

252/794

252/793

510/178

252/18628

252/792

510/407

134/42

510/376

510/313

510/302

510/276

510/516

510/308

252/1862

438/745

252/18625

510/466

510/481

510/469

510/501

424/7031

424/7017

510/456

8/137

424/7016

424/7011

510/443

8/1157

424/7013

8/1275

8/12751

510/521

510/350

134/13

510/500

510/367

510/301

510/306

510/307

510/298

510/322

510/324

510/330

510/331

510/332

510/264

510/417

510/182

510/439

510/311

510/356

510/513

510/257

510/375

510/474

510/471

510/312

554/1
 510/506
 510/470
 510/505
 510/437
 510/255

-----keywords-----

peroxytetramethylammonium peroxytetraethyl peroxytetrachloride dmso diamine quaternary ammonium carb oxylate semiconductor quaternary ammonium hydrogen peroxide tetramethyl tetraethyl ammonium tetr aethylammonium hydroxide tetramethylammonium hydroxide photoresist resist residue residue titanium oxide titanium stripping dimethyl sulfoxide dimethyl glycol ether amide organic solvent soluble organic organic peracid organic percarbonate perborate persulfate hydrogen peroxide tetraalkyl ammonium phthalic acid phthalic salicylic acid salicylic benzoic acid benzoic aromatic carboxylic aromatic carboxylic acid carboxylic carboxylate ammonium carbonate ammonium hydroxide hydroxide ammonium salt quaternary ammonium quaternary peroxide resist stripper resist peroxycarboxylate tetraalkyl benzyltrialkyl alkyl-hydroxylalkyl peracetic hydroperoxide peroxyhydrate sulfoxide circuit sulfone lactam imidazolidinone ammonium oxide salt acid hydrogen amine soluble ether glycol solvent tetramethylammonium tetraethylammonium disclosures distilled dropwise heated Tertiary Ammonium persulfate adipate propionate lactate benzoate salicylate Severe aqueous hydrogen copper aluminum aluminum copper washed immersed plasma ashing mask silicon wafer wafer positive photoresist immersion ultrasonic waves ultrasonic photoresist residue additive oxidized metal salt enzyme sucrose glucose sugar phenol hydroxyl diethylenetriamine ptaacetic acid ethylenediaminetetraacetic acid ethylenediaminetetraacetic aminopolycarboxylic acid formic acid formic organic solvents solvents monobutyl ether glycol monobutyl monobutyl monoethyl ether glycol monoethyl monoethyl monomethyl ether glycol monomethyl monomethyl glycol ethers ethers tripropylene glycol tripropylene glycol dipropylene glycol dipropylene propylene glycol propylene triethylene glycol triethylene diethylene glycol diethylene ethylene glycol glycols amides amines corrosion diamines ethylenediamine morpholine ethanolamine ethanol triethanolamine diethanolamine monoethanolamine alkanolamines benzyl moiety propionic acid propionic adipic acid adipic lactic acid lactic acid acetic aliphatic carboxylic aliphatic ammonium salts alkalinity aryl alkyl benzoyl peracetic acid metal ions potassium salt potassium sodium salt ammonia crystallization urea anhydride alkaline oxides dissolved phase species insoluble titanium dioxide solubilized alkali acidic dissolve decomposition unstable ion implantation

-----references-----

----- 6323169
 classes:1 510/176 1 510/175 1 510/245 1 510/255 1 510/257 1 510/264 1 510/499
 score: 1005

keywords: semiconductor;tetramethyl;tetramethylammonium hydroxide;photoresist;resist residue;residue;titanium;stripping;dimethyl;amide;organic solvent;organic;hydroxide;quaternary;resist;circuit;sulfone;ammonium;oxide;salt;acid;hydrogen;amine;ether;glycol;solvent;tetramethylammonium;aluminum;copper;immersed;mask;ultrasonic;oxidized;diethylenetriaminepentaacetic acid;ethylenediaminetetraacetic acid;ethylenediaminetetraacetic;aminopolycarboxylic acid;organic solvents;solvents;glycol monobutyl;monobutyl;glycol monoethyl;monoethyl;monomethyl ether;glycol monomethyl;monomethyl;dipropylene glycol;dipropylene;propylene glycol;propylene diethylene glycol;diethylene;ethylene glycol;corrosion;ethylenediamine;monoethanolamine;ammonium salts;alkaline;oxides;alkali;acidic;dry etching;inorganic;alkanolamine;resist stripping;etching;exposure;integrated circuits;semiconductor integrated;semiconductor;tetramethylammonium hydroxide;photoresist;resist residue;residue;titanium;stripping;dimethyl;amide;organic solvent;organic;hydroxide;quaternary;Resist;Inventors;

- is usually carried out at a temperature from ordinary temperature to 80.degree. C., and the specific stripping temperature may be appropriately selected depending upon etching conditions and materials for inorganic substrate used.

Materials for the inorganic substrates may be semiconductive circuit-forming materials such as silicon, amorphous silicon, polysilicon, silicon oxide film, silicon nitride film, **aluminum**, **aluminum** alloys, **copper**, **copper** alloys, **titanium**, **titanium**-tungsten, **titanium** nitride, tungsten, tantalum, tantalum compounds, tantalum alloys, chromium, chromium oxide, chromium alloys and ITO (indium-tin oxide); compound **semiconductor**s such as gallium-arsenic, gallium-phosphorus and indium-phosphorus; and glass substrates for LCD.

The stripping process of the present invention is used to remove resist films coated onto the inorganic substrate, patterned resist films remaining after etching, or resist residues rem

- plasma asking. It was obtained by disposing the Al alloy (Al--Cu) film for circuit pattern on the silicon substrate. On the Al alloy film, a resist composition was coated to form a resist film, which was then patterned by photolithography. Then dry etching with

a fluorine-based gas was carried out using the patterned resist film as a mask to make the Al alloy film to circuit pattern, followed by oxygen **plasma ashing**. In FIG. 1, the resist residues remained on side walls of the Al alloy circuit pattern.

The Al alloy circuit device was **immersed** in a resist stripping composition having chemical composition as shown in Table 1 under respective conditions, rinsed with ultrapure water and then dried. Then, the surface of the circuit device was observed under scanning electron microscope (SEM) to evaluate the removal of the resist residues and the corrosion of the Al alloy according to the following ratings. The results are shown in

----- 6492311
classes:1 510/176 1 510/175 1 510/245 1 510/255 1 510/477 1 510/488 1 510/499
score: 682

keywords: dmsos;semiconductor;photoresist;residue;titanium;stripping;dimethyl;organic solvent;organic;ammonium salt;resist;circuit;ammonium;oxide;salt;acid;amine;soluble;glycol;solvent;disclosures;aluminum;immersed;silicon wafer;wafer;ethylenediaminetetraacetic acid;ethylenediaminetetraacetic;solvents;propylene glycol;propylene;triethylene glycol;triethylene;diethylene glycol;diethylene;ethylene glycol;amines;corrosion;alkanolamines;ammonium salts;alkyl;ammonia;decomposition;inorganic;alkanolamine;etching;integrated circuit;integrated circuits;semiconductor;photoresist;residue;titanium;stripping;dimethyl;organic solvent;organic;ammonium salt;Resist;

- ing composition of the present invention for a time and at a temperature sufficient to remove the residue. The substrate is generally **immersed** in the **stripping** and cleaning composition. The time and temperature are determined based on the particular material being removed from a substrate. Generally, the temperature is in the range of from about ambient or room temperature to about 120.degree. C. and the contact time is from about 2 to 60 minutes.

The substrate may then be rinsed in a polar solvent, such as isopropyl alcohol, followed by a deionized water rinse. The substrate is then mechanically dried, such as with a spin drier, or nitrogen blow dried.

The following represent non-limiting examples and describe the invention further.

Examples of cleaning compositions according to the present invention suitable for removing resist or other organic residues from a substrate are set forth in Table I below.

TABLE 1

- lations. The wafers were broken into pieces before and after processing in these different cleaning solutions and subsequently viewed under a FE Hitachi 4500 Scanning Electron Microscope for post ash residue removal in vias and substrate compatibility on metal stacks.

EXAMPLE 1

A via opening with a size of 1.2 micron in a silicon oxide dielectric layer was etched through a **photoresist** patterned opening using a standard silicon oxide plasma etching process. The **photoresist** was removed by oxygen **plasma ashing**. FIG. 1A is a micrograph of a scanning electron microscope (SEM) image for a representative substrate of the type used in this example, showing that heavy organometallic etch residue remained on the substrate surface, particularly around the via opening. The substrate was then processed in composition A for 30 minutes at 55.degree. C. FIG. 1B, the resulting SEM photograph, shows that composition A removed all the organ

- um metallurgy. FIG. 3B shows complete cleaning of a TiN/Al-Cu/Ti/TiN/Ti stack wafer, with no metal corrosion, or just starting to attack the **aluminum** layer in the stack metallurgy.

EXAMPLE 4

The procedures of Example 3 were repeated with composition C, also at a cleaning temperature of 45.degree. C. FIGS. 4A and 4B show the results obtained. Complete cleaning of the ashed via wafer was obtained, as determined by visual observation of FIG. 4A. There was complete control of metal corrosion. FIG. 4B shows complete cleaning of the TiN/Al-Cu/Ti/TiN/Ti stack wafer, as shown by the clean appearance of the top TiN layer with no metal corrosion.

EXAMPLE 5

The procedures of Example 3 were repeated with composition D but at a cleaning temperature of 65.degree. C. FIGS. 5A, 5B and 5C show the results obtained. A 90% cleaning of the ashed via wafer was obtained, as determined by visual observation of FIG. 5A. There was no metal corrosion.

6524376
classes:1 106/1442 1 106/1441 1 106/1443 1 106/1444 1 252/791 1 252/394 1 438/692
score: 582

keywords: semiconductor;hydrogen peroxide;peroxide;residue;stripping;dimethyl sulfoxide;dimethyl;organic solvent;organic;hydrogen peroxide;salicylic;benzoic;aromatic;carboxylic acid;carboxylic;hydroxide;peroxid e;resist;stripper;sulfoxide;oxide;salt;acid;hydrogen;soluble;ether;glycol;solvent;Severe;aluminum;copper;immersed;plasma ashing;silicon wafer;wafer;ultrasonic;oxidized;hydroxyl;monobutyl ether;glycol monobutyl;monobutyl;monoethyl ether;glycol monoethyl;monoethyl;monoethyl ether;glycol monomethyl;monomethyl;diethy lene glycol;diethylene;ethylene glycol;amides;corrosion;monoethanolamine;propionic;lactic;acetic;alkyl;am monia;urea;alkali;decomposition;dry etching;alkanolamine;etching;exposure;wiring;semiconductor;residue;st ripping;dimethyl sulfoxide;dimethyl;organic solvent;organic;hydrogen peroxide;salicylic;benzoic;aromatic; carboxylic acid;carboxylic;hydroxide;peroxide;Resist;stripper;Inventors;

- which was a positive resist material for KrF. The resist film was exposed to a light via a mask pattern, followed by development with an aqueous tetramethylammonium **hydroxide** solution, to obtain a resist pattern.

Using this resist patter as a mask, the silicon oxide film was dry-etched until the silicon nitride film was exposed, to form through holes having a diameter of 0.2 .mu.m. As the etching gas, a fluorocarbon type gas was used. After the completion of the etching, oxygen **plasma ashing** was conducted to remove part of the resist pattern. Thereafter, a peeling treatment was conducted using a stripper composition shown in No. 1 of Table 4.

Next, the silicon nitride film was etched using a different etching gas, to expose each **copper** wiring at the bottom of each through hole. In order to remove the etching **residue** generated in the above etching, a peeling treatment was conducted again using the same stripper composition (No.

- 1 in Table 4) as used above.

The same procedure as above was conducted using each of stripper solutions Nos. 2 to 8 shown in Table 4, to produce total 8 kinds of samples.

Each sample (wafer) obtained above was rinsed with pure water, and its section was observed by a scanning electron microscope (SEM) to examine (1) **stripping** ability of resist film and etching **residue** and (2) corrosion inhibitability for **copper** film. The following rating standards were used.

Stripping Ability

The degree of remaining of resist film and etching **residue** was examined visually and rated by the following three grades.

.largecircle.: Substantially no remaining

.DELTA.: Remaining in a small amount

x: Remaining in a large amount

Corrosion Inhibitability

The corrosion status of **copper** film surface was examined visually and rated by the following four grades.

.circleincircle.: **copper** film had no corrosion.

.largecircle.: **copper** film had slight corrosion.

.DELTA.: **copper** film ha

- .smallcircle.

x

7 Gallic acid (5) NMEA (60) Remainder .smallcircle.

x

8 Sorbitol (5) NMEA (60) Remainder .smallcircle.

x

Urea (25)

*1 In the column of water, "remainder" refers to a remainder after the amounts of anticorrosive agent and peeling agent have been subtracted from 100% by mass.

*2 NMEA: N-methylethanolamine

MEA: monoethanolamine

As is clear from the above, each stripper composition using an anticorrosive agent of the present invention has excellent **stripping** ability and corrosion inhibitability. In the present example, the present invention was applied to a single damascene process; however, the present invention can also be applied to a dual damascene process.

EXAMPLE 5

A silicon wafer having a **copper** film on the whole surface was **immersed** in various given stripper solutions at 80.degree. C. for 10 minutes. The etching rate of **copper** in eac

6264839
classes:1 210/687 1 210/699 1 210/700 1 525/340 1 525/383 1 526/277 1 526/278 1 526/310 1 526/312
score: 553

keywords: peroxy;hydrogen peroxide;peroxide;dimethyl;glycol ether;amide;organic;perborate;hydrogen peroxide;phthalic;salicylic;carboxylic;carbonate;hydroxide;quaternary;peroxide;peracid;hydroperoxide;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;glycol;distilled;dropwise;heated;copper;washed;glucose;sugar;diethylenetriamine;pentaaacetic acid;ethylenediaminetetraacetic acid;ethylenediaminetetraacetic;formic acid;formic;solvents;ethers;propylene;ethylene glycol;glycols;amides;amines;corrosion;ethanolamine;benzyl;moiety;lactic;acetic;aliphatic;ammonium salts;alkyl;metal ions;potassium;sodium salt;ammonia;anhydride;alkaline;oxides;dissolved;insoluble;alkali;dissolve;decomposition;inorganic;dimethyl;glycol ether;amide;organic;perborate;hydrogen peroxide;phthalic;salicylic;carboxylic;carbonate;hydroxide;quaternary;peroxide;

- d
tert-butyl perpivalate is added after 1/4 hour, 11/4 hours and 2 hours in a 2 g portion each time. The thin suspension of the copolymer is concentrated under a waterjet vacuum in a rotary evaporator until dry. The K value of the polymer obtained as a slightly brownish powder is, measured in 1% strength in cyclohexanone, 9.0.

Polymer 13

In a reactor as described for the preparation of polymer 1, 350 g of water, 117.5 g of maleic anhydride, 6.45 g of ferroammonium sulfate and 86.4 g of sodium **hydroxide** are **heated** to the boil and then admixed in the course of 5 hours with a solution of 232 g of acrylic acid and 41 g of water and also a solution of 93 g of N-vinylformamide and 27 g of water and 237 g of 30% strength sodium **hydroxide** solution and in the course of 6 hours with a solution of 46 g of **hydrogen **peroxide** in 50 g of water added at uniform rates. This is followed by a further 2 hours of heating at the boil. The pH 0

- f
the resulting solution is 5.1, and the K value of the resulting polymer, measured in 1% strength in water, is 42.

Polymer 14

In a reactor as described for the preparation of polymer 1, 450 g of water, 50 g of vinylpyrrolidone, 0.05 g of sodium **hydroxide**, 1 g of N,N'-divinylethyleneurea and 0.1 g of sodium dithionite are **heated** at 60.degree. C. and [sic] a slow stream of nitrogen. After about 45 minutes, white polymer flakes precipitate from the previously clear solution, gradually increasing in number. After 10 minutes, a solution of 150 g of N-vinylformamide and 3 g of N,N-divinylethyleneurea is metered in over 20 minutes and stirred and the whole is stirred at 60.degree. C. over 2 hours. During this period the polymer solution must be diluted with 1250 g of water to maintain stirrability. After cooling, the viscous polymer is filtered off, **washed** with 5 l of water and dried at 50.degree. C. in a vacuum drying cabinet. The y complete phosphonomethylation is

- desired. The mixture is then refluxed. An aqueous, approximately 30% strength formaldehyde solution is slowly added **dropwise**. The molar ratio of formaldehyde to VFA originally present in the polymer is within the range from 4:1 to 8:1 (cf. Table 1).

On completion of the addition the mixture is refluxed for a further 15 hours. The volatiles and excess reagents are removed by a 2-hour steam treatment, and the solution obtained is concentrated to about a third of its volume.

To purify the phosphonomethylated polymers, the polymers are precipitated in the form of the sodium salt by adding the still acid concentrated solution **dropwise** to ten times the volume of methanolic sodium **hydroxide** solution in which the amount of NaOH used should correspond to the acid number equivalent of the concentrated reaction mixture. If the sodium salts are obtained as solids, they are filtered off, **washed** with methanol

4105783
classes:1 514/459
score: 538

keywords: amide;organic;salicylic;ammonium hydroxide;hydroxide;ammonium salt;ammonium;salt;acid;amine;solvent;distilled;Tertiary;Severe;propylene;amides;amines;diamines;ethylenediamine;ethanolamine;triethanolamine;diethanolamine;monoethanolamine;lactic acid;lactic;ammonium salts;alkyl;ammonia;anhydride;dissolved;inorganic;amide;organic;salicylic;ammonium hydroxide;hydroxide;ammonium salt;

- seasons, when environmental humidity is comparatively low. They are characterized by fissures, chaps, cracks or flakes of the skin on hands, face, neck and legs.

Conventional treatments for all kinds of dry skin conditions primarily involve the topical application of oils or oil preparations, and hydrating emollients. In addition, ointments containing **salicylic acid**, urea, glycerol, propylene glycol, sorbitol or vitamin A have been used. Prior treatments, however, have not been universally successful, and have, in many cases, been unable to promote healing to cause a complete remission of the symptoms. Because the mechanisms involved in causing dry skin are not known, treatment has usually resulted in a temporary remission or healing of the flaky or scaly lesions.

We have now discovered that "dry skin" conditions may be successfully prevented or treated with the acid, amide or **ammonium salt** of .alpha.- or .beta.-hydroxyac

- butyric acid.

Generally, the amide may be formed from acid anhydride or lactone and ammonia or any organic primary or secondary amine. The **ammonium salt** may be formed directly from acid and an organic primary, secondary or **Tertiary** amine.

Preferred organic primary amines include any alkylamines such as methylamine and ethylamine; ethanolamines such as monoethanolamine and monoisopropanolamine; and **diamine**s such as ethylenediamine and 1,2-diaminopropane.

Preferred organic secondary amines include dialkylamines such as **dimethyl**amine and diethylamine; diethanolamine and diisopropanolamine; N-methylethanamine and N-ethylethanamine.

Preferred organic **Tertiary** amines include trialkylamines such as trimethylamine and triethylamine; triethanolamine; N-methyldiethanolamine and triisopropanolamine.

It has been established through tests on humans having "dry skin" conditions that topical application of a lotion, cream or ointment

- ss. In treatment of common dry skin conditions according to this invention we found that the above composition with low pH could cause some skin irritation (redness and sensation of burning) on some of the sensitive subjects. It was therefore desirable to develop compositions which were therapeutically effective but not irritative.

Most inorganic alkalis, forming inorganic salts with .alpha.- or .beta.-hydroxyacids or .alpha.-ketoacids that do not readily penetrate human skin, cannot be used to neutralize these acids. It has previously been discovered that certain organic bases, and **ammonium **hydroxide**, may be successfully used to raise the pH of the compositions containing .alpha.- or .beta.-hydroxy acids or .alpha.-ketoacids without compromising the therapeutic efficaciousness of the active ingredients. Under such conditions the active ingredients are in the form of amide or ammonium salt. The organic bases may include any or

----- 6410494
 classes:1 510/175 1 134/13 1 134/2 1 134/3 1 134/41 1 438/745 1 510/177
 score: 526

keywords: semiconductor;peroxide;organic solvent;organic;phthalic;salicylic;peroxide;circuit;ammonium;oxide;acid;amine;solvent;washed;ultrasonic;hydroxyl;aminopolycarboxylic acid;formic;amines;corrosion;ethylenediamine;propionic;adipic acid;adipic;lactic acid;lactic;acetic acid;acetic;potassium;oxides;dissolved;alkali;dissolve;ion implantation;inorganic;wiring;semiconductor;organic solvent;organic;phthalic;salicylic;peroxide;Inventors;

-	10.sup.11	1 .times.	10.sup.10	2 .times.	10.sup.11			
15	Citric acid	5	NTPO		0.5	1 .times.		
10.sup.11	2 .times.	10.sup.10	4 .times.	10.sup.11				
16	Malonic acid	5	NTPO		0.5	4 .times.		
10.sup.11	4 .times.	10.sup.11	7 .times.	10.sup.11				
17	Succinic acid	5	NTPO		0.5	5		
.times.	10.sup.11	5 .times.	10.sup.11	7 .times.	10.sup.11			
18	Acetic acid	5	NTPO		0.5	6 .times.		
10.sup.11	9 .times.	10.sup.11	1 .times.	10.sup.12				
19	Glutaric acid	1	Potassium		5	2		
.times.	10.sup.11	5 .times.	10.sup.11	4 .times.	10.sup.10			
					thiocyanate			
20	Citric acid	20	EDDPO		1	4 .times.		

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10.sup.10 2 .times. 10.sup.11 1 .times. 10.sup.12
21 Adipic acid 1 Ammonium 10 8 .time
- efficiency higher than
that shown by acetic acid which is an organic acid having only one
carboxyl group.

EXAMPLE 3

Each one of silicon wafers provided with wirings of Al and Cu on the substrate surface thereof was dipped in the cleaning agent of the present invention, having the formations of Nos. 1 and 2 as shown in Table 1, respectively relating to Example 1 as mentioned above, at 70.degree. C. for 1 hour. After that, each one of thus obtained silicon wafers was **washed** with ultra-pure water and dried by use of a spin-dryer. The metallized wirings on the substrate surface were confirmed by a microscopic observation, and further confirmed by a circuit tester whether there is disconnection or not. As the result, it was confirmed that neither corrosion of the metallized wirings nor disconnection was observed on the surface of the silicon wafers which was dipped in the cleaning agent of the present invention.

Reference Examp

----- 4197316
classes:1 514/554 1 514/459 1 514/460 1 514/471 1 514/473 1 514/546 1 514/553 1 514/557 1 514/570 1 514/574 1 514/613 1 514/617 1 514/663 1 514/667 1 514/669 1 514/673
score: 526

keywords: amide;organic;salicylic;ammonium hydroxide;hydroxide;ammonium salt;ammonium;salt;acid;amine;glycol;solvent;distilled;Tertiary;Severe;propylene glycol;propylene;amides;amines;diamines;ethylenediamine;ethanolamine;diethanolamine;monoethanolamine;lactic acid;lactic;ammonium salts;alkyl;ammonia;anhydride;disolved;inorganic;amide;organic;salicylic;ammonium hydroxide;hydroxide;ammonium salt;

- hydrating emollients. In addition, ointments containing **salicylic acid**, urea, glycerol, propylene glycol, sorbitol or vitamin A have been used. Prior treatments, however, have not been universally successful, and have, in many cases, been unable to promote healing to cause a complete remission of the symptoms. Because the mechanisms involved in causing dry skin are not known, treatment has usually resulted in a temporary remission or healing of the flaky or scaly lesions.

We have now discovered that "dry skin" conditions may be successfully prevented or treated with the acid, **amide** or **ammonium salt** of .alpha.- or .beta.-hydroxyacids or .alpha.-keto acids and esters thereof. The compounds of the present invention include citric acid, glycolic acid, glucuronic acid, galacturonic acid, glucuronolactone, gluconolactone, .alpha.-hydroxybutyric acid, .alpha.-hydroxyisobutyric acid, lactic acid, malic acid, mandelic acid, mucic acid, pyruvic acid, methylpyruvate, ethyl pyruvate, .beta.-phenylactic acid, .beta.-phenylpyruvic acid, saccharic acid, tartaric acid, tartronic acid, and .beta.-hydroxybutyric acid. Generally, the **amide** may be formed from acid anhydride or lactone and ammonia or any organic primary or secondary amine. The **ammonium salt** may be formed directly from acid and an organic primary, secondary or **Tertiary** amine.

Preferred organic primary amines include any alkylamines such as methylamine and ethylamine; ethanolamines such as monoethanolamine and monoisopropanolamine; and diamines such as ethylenediamine and 1,2-diaminopropane.

Preferred organic secondary amines include dialkylamines such as **dimethyl**amine and diethylamine; diethanolamine and diisopropanolamine; N-methylethanamine and N-ethylethanamine.

Preferred organic **Tertiary** amines include trialkylamines such as trimethylamine and triethylamine; triethanolamine; N-methyldiethan-

- y effective but not irritative.

Most inorganic alkalis, forming inorganic salts with .alpha.- or .beta.-hydroxyacids or .alpha.-ketoacids that do not readily penetrate human skin, cannot be used to neutralize these acids. It has previously been discovered that certain organic bases, and **ammonium **hydroxide**, may be successfully used to raise the pH of the compositions containing .alpha.- or .beta.-hydroxy acids or .alpha.-ketoacids without compromising the therapeutic efficaciousness of the active ingredients. Under such conditions the active ingredients are in the form of **amide** or ammonium salt. The organic bases may include any organic amine of primary, secondary or **Tertiary** family. The organic primary amines may include any

alkylamines such as methylamine and ethylamine; any ethanolamines such as monoethanolamine and monoisopropanolamine; any diamines such as ethylenediamine and 1,2-diaminopropane. The organic secondary amine

----- 5994290

classes:1 510/531 1 510/276 1 510/300 1 510/302 1 510/305 1 510/310 1 510/315 1 510/318 1 510/323 1 510/3
40 1 510/361 1 510/367 1 510/372 1 510/374 1 510/377 1 510/392 1 510/398 1 510/434 1 510/443 1 510/480 1
510/507 1 510/509 1 510/532 1 510/533
score: 517

Keywords: hydrogen peroxide;peroxide;amide;organic;percarbonate;perborate;hydrogen peroxide;phthalic;benz oic;aromatic;carboxylic acid;carboxylic;carbonate;peroxide;sulfone;ammonium;oxide;acid;hydrogen;amine;sol uble;ether;glycol;Tertiary;propionate;washed;oxidized;enzyme;sucrose;hydroxyl;ethers;propylene glycol;pro pylene;ethylene glycol;glycols;amines;aliphatic;ammonium salts;alkyl;metal ions;potassium;urea;anhydride; alkaline;oxides;species;insoluble;alkali;inorganic;amide;organic;percarbonate;perborate;hydrogen peroxide; phthalic;benzoic;aromatic;carboxylic acid;carboxylic;carbonate;peroxide;

- xyclic acid or monoperoxophthalic acid, adducts of hydrogen **peroxide** onto inorganic salts, eg. sodium perborate monohydrate, sodium perborate tetrahydrate, sodium **carbonate** perhydrate or sodium phosphate perhydrate, adducts of hydrogen **peroxide** onto organic compounds, eg. urea perhydrate, or of organic peroxy salts, eg. alkali metal **persulfate**s or peroxodisulfates, where appropriate in combination with from 0 to 15% by weight, preferably 0.1 to 15% by weight, in particular 0.5 to 8% by weight, of bleach activators (G). In the case of color detergents, the bleach (F) is (when present) normally used without bleach activator (G), otherwise bleach activators (G) are usually also present.

Suitable bleach activators (G) are:

polyacylated sugars, eg. pentaacetylglucose;
acycloxybenzenesulfonic acids and their alkali metal and alkaline earth metal salts, eg. sodium p-isobornanoyloxybenzenesulfonate or sodium p-benzoxybenzenesulfonate;

N,N-

----- 4338204

classes:1 510/301 1 510/306 1 510/307 1 510/308 1 510/322 1 510/324 1 510/330 1 510/331 1 510/332
score: 484

Keywords: peroxy;diamine;quaternary ammonium;dimethyl;organic;percarbonate;perborate;quaternary ammonium; quaternary;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;Tertiary;washed;additive;enzyme;phenol;e thers;diethylene;glycols;amines;benzyl;lactic;aliphatic;alkyl;potassium;crystallization;anhydride;alkalin e;insoluble;alkali;inorganic;dimethyl;organic;percarbonate;perborate;quaternary ammonium;quaternary;

- e in the range 85.degree.-90.degree. C. and spray drying the slurry to give a granular base powder. The order of addition of ingredients was anionic surfactant, silicate, minor ingredients, sulphate, nonionic, clay, cationic and finally phosphate. The copolymer of Example 3 was added with the CMC. Inlet air temperatures in the range 320.degree. C. to 340.degree. C. were used and the spray dried granules were subsequently cooled to 25.degree.-35.degree. C. in an air lift using ambient air as the cooling medium. The heat sensitive solid ingredients (c) were then added to the base powder through feeding devices known to those skilled in the art and the granule mix was subjected to a spray-on of the **Tertiary** amine component (b) into which perfume material has been blended.

EXAMPLES 4-11

Further compositions in accordance with the invention are given below:

-- 20.0	
25.0	
-- 20.0	
20.0	
-- 21.1	
Silica-Silicone suds	
suppressor 0.2	
-- 0.2	
0.2	
-- 0.2	
0.2	

1.2

Hydrophobic silica-	
mineral oil-wax suds	
suppressor -- 0.2	-- -- 0.2
-- -- --	
Tetra sodium ethylene	
diamine tetra methylene	
phosphonate -- 0.5	0.5
	-- 0.5
	0.5
-- --	
Maleic anhydride-	
methyl vinyl ether	
copolymer -- 0.5	1.0
	-- 0.5
	0.5
-- --	
Stearic Acid 0.	

----- 4238192
 classes:1 8/111 1 252/18628 1 252/18629 1 252/18643
 score: 484

keywords: diamine;hydrogen peroxide;peroxide;tetramethyl;organic;hydrogen peroxide;phthalic acid;phthalic ;benzoic;aromatic;carboxylic acid;carboxylic;peroxide;peracid;ammonium;oxide;acid;hydrogen;amine;heated;T ertiary;phenol;formic;diethylene;amides;adipic acid;adipic;lactic;acetic;aliphatic;aryl;alkyl;dissolved;s pecies;decomposition;unstable;organic;hydrogen peroxide;phthalic acid;phthalic;benzoic;aromatic;carboxyli c acid;carboxylic;peroxide;

- casionally an acid is not required to bring the composition to within the range of 1.5-5.5, however, it is generally necessary and in fact preferred to incorporate at least one acid into the composition.

As used in this specification and in the attached claims the term "organic acid" means **carboxylic acid**'s wherein the acid functionality predominates over other groups present in the compound. This term does not include alpha-amino monocarboxylic acids but does include tetra and penta **carboxylic acid**'s containing some nitrogen.

Suitable **organic** acids include various saturated and unsaturated mono-, di-, tri- tetra- and pentacarboxylic acids, such as acetic acid, oxalic acid, formic acid, adipic acid, maleic acid, tartaric acid, lactic acid, gluconic acid, glucaric acid, glucuronic acid, ascorbic acid; mono- and dicarboxylic acids of benzene such as **benzoic acid**, **phthalic acid** and substituted **aromatic** acids and salts of these carb

----- 4292035
 classes:1 8/137 1 510/516 1 510/521
 score: 477

keywords: diamine;quaternary ammonium;stripping;dimethyl;glycol ether;organic;carboxylic;carbonate;hydrox ide;quaternary ammonium;quaternary;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;glycol;Tertiary; washed;phenol;ethers;triethylene glycol;triethylene;ethylene glycol;amides;amines;diamines;aliphatic;alky l;metal ions;potassium salt;potassium;sodium salt;alkaline;dissolved;insoluble;alkali;inorganic;stripping ;dimethyl;glycol ether;organic;carboxylic;carbonate;hydroxide;quaternary ammonium;quaternary;

- um chloride -- -- 4.5	
Branched C.sub.12 alkylbenzene	
sulfonate, Na salt	-- 4.9 9.1
	-- 0.5 0.9
Sodium toluenesulfonate	-- 15.5 --
Sodium **carbonate** --	3.4 6.4
Sodium Sulfate --	-- 37.7
Sodium Sesquicarbonate	
(anhyd. basis) -- --	12.5 10.0
Water --	Bal. Bal.
Miscellaneous --	

The results after two wash-rinse cycles are:

Blank	#2	#6
0-	+3.2	+2.9

Least significant difference at 95% confidence=0.5

Composition #2 was prepared in slurry form as described in Example I.

Composition #6 was prepared as follows:

2.25 g. of ditallodimethylammonium chloride was dispersed in tap water at 70.degree. C. T

- mperature

70.degree. F.

Water hardness 9 grains/gal. 3:1 Mg:Ca ratio

Water:cloth ratio

20:1

Washing time 20 minutes

After washing, the test swatches were hand wrung and rinsed, with agitation, for 5 minutes in the mini-washer using 1 gallon of 70.degree. F. water as above.

One-half of the test swatches were rinsed a second time following the procedure used in the first rinse. The other one-half of the test swatches were similarly rinsed a second time with the exception that the second rinse water included 3.78 grams of the fabric softening composition recited in Example I No. 1. All the test swatches were then hand wrung and line dried.

The test swatches were then visibly stained with dirty motor oil (DMO) or bacon grease and allowed to age for about 18 hours.

The test swatches of each soil type were **washed** separately using the washing con

----- 6365564
 classes:1 510/376 1 510/276 1 510/302 1 510/312 1 510/313 1 510/356 1 510/367 1 510/375 1 510/421 1 510/43 1 510/456 1 510/501
 score: 473

keywords: peroxy;diamine;hydrogen peroxide;peroxide;residue;organic;percarbonate;perborate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;quaternary;peroxide;peracid;peroxyhydrate;lactam;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;solvent;disclosures;aluminum;washed;additive;enzyme;sugar;phenol;diethylenetriaminepentaacetic acid;ethylenediaminetetraacetic acid;ethylene diaminetetraacetic;solvents;propylene;amides;ethylenediamine;moiety;acetic;aliphatic;ammonium salts;alkalinity;aryl;alkyl;metal ions;sodium salt;urea;anhydride;alkaline;dissolved;species;alkali;acidic;inorganic;residue;organic;percarbonate;perborate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;quaternary;peroxide;

- 1 1 0
 Benzotriazole 0.1 0.1 0.1 0
 Sodium Sulfate, water, minors BALANCE 100% 100% 100% 100%

TO:

Note 1: Bleach Activator according to Example 1.

Note 2: These **hydrogen **peroxide** sources are expressed on a weight % available oxygen basis. To convert to a basis of percentage of the total composition, divide by about 0.15.

Note 3: Transition Metal Bleach Catalyst: Pentaamineacetatocobalt (III) nitrate; may be replaced by MnTACN.

EXAMPLE VI

Cleaning compositions having liquid form especially useful for cleaning bathtubs and shower tiles without being harsh on the hands are as follows:

Ingredient	% (wt.)	
	A	B
Bleach Activator*	7.0	5.0
hydrogen **peroxide	10.0	10.0
C.sub.12 AS, acid form, partially neutralized	5.0	5.0
C.sub.12-14		

----- 4642197
 classes:1 510/298 1 252/1862 1 252/18625 1 510/313 1 510/350 1 510/439 1 510/469 1 510/470 1 510/471 1 510/474 1 510/475 1 510/513

score: 463

keywords: quaternary ammonium;residue;organic;perborate;quaternary ammonium;quaternary;ammonium;oxide;salt;acid;amine;aluminum;washed;additive;metal salt;glucose;sugar;ethers;ethylenediamine;propionic;alkyl;sodium salt;dissolved;alkali;dissolve;residue;organic;perborate;quaternary ammonium;quaternary;

- toms as the long-chain C.sub.10 -C.sub.20 alkyl or alkenyl radical. For the rest, suitable **quaternary** ammonium compounds contain three identical or different C.sub.1 -C.sub.4 alkyl groups. These short-chain alkyl groups are, in particular, methyl groups. One particularly suitable and, therefore, preferred surface-active **quaternary ammonium** compound is tetradecyl trimethylammonium bromide. The **quaternary ammonium** compound may be completely or partly replaced by one or more surface-active fatty amine compounds.

Advantageously the fatty amine compounds are fatty amine lower alkoxylates, preferably fatty amine ethoxylates derived from C.sub.10 -C.sub.20 fatty amines. These compounds are preferably adducts of from 1 to 6 mols of ethylene oxide with 1 mol of a primary fatty amine containing a long-chain (C.sub.10 -C.sub.16) alkyl or alkenyl radical. One example of a particularly suitable fatty amine ethoxylate is the adduct of 2 mol

- ing auxiliaries. The powder density of the granulate sifted through a 2 mm mesh sieve amounts to between 600 and 700 g per liter.

The granulate coated with a protective layer and dried is preferably mixed with from 10 to 20% by weight, based on the granulate, of a tablet disintegrating agent, preferably a mixture of two different tablet disintegrating agents, more especially a mixture of crosslinked polyvinyl pyrrolidone and/or cellulose ethers and/or swellable magnesium **aluminum** silicate. The effect of a single tablet disintegrating agent may be enhanced by the addition of up to 7% by weight (based on the weight of the tablet) of an alkali metal salt of short-chain organic mono- or polycarboxylic acids, for example sodium acetate or sodium citrate. In the case of the preferred addition of a tablet disintegrating agent mixture containing polyvinyl pyrrolidone, a ratio by weight of polyvinyl pyrrolidone to the second table

- a breaking strength of 10 kg which was determined as follows:

In a Chatillon Tension and Compression Tester, the tablet was placed on a cavity block with a cavity somewhat smaller than the diameter of the tablet. The cavity block was moved by motive force on an anvil against a dynamometer with a wedge-shaped cutting edge. The movement was continued until the tablet broke. The force read off on breakage of the tablet is the breaking strength in kg. This method of determining breaking strength is described in detail in W. A. Ritschel's book entitled "Die Tablette", Edition Cantor KG, Aulendorf in Wuerttemberg, 1960, pages 312 and 313.

The tablets obtained dissolved completely in 6 minutes in a 30.degree. C. wash (carried out in a Miele type 430 automatic washing machine). Despite two thirds of the recommended dosage of an inexpensive detergent, the soiled laundry **washed** in the presence of the tablets was distinctly cleaner than

----- 6479448

classes:1 510/296 1 510/325 1 510/337 1 510/342 1 510/343 1 510/356 1 510/421
score: 446

keywords: diamine;quaternary ammonium;titanium;dimethyl;organic solvent;organic;percarbonate;perborate;per sulfate;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;ammonium hydroxide;hydroxide;quaternary ammonium;quaternary;tetraalkyl;peroxyhydrate;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;glycol;solvent;heated;copper;ultrasonic;enzyme;sugar;phenol;hydroxyl;solvents;monobutyl ether;glycol monobutyl;monobutyl;dipropylene glycol;dipropylene;diethylene glycol;diethylene;glycols;amines;ethanolamine;ethanol;triethanolamine;monoethanolamine;moiety;aliphatic;ammonium salts;alkyl;potassium;alkaline;oxides;phase;species;insoluble;alkali;dissolve;inorganic;titanium;dimethyl;organic solvent;organic;percarbonate;perborate;persulfate;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;ammonium hydroxide;hydroxide;quaternary ammonium;quaternary;tetraalkyl;

- peroxygen bleaching agents may also be used in particulate form in the detergent compositions herein.

Inorganic bleaching agents are in fact preferred. Such inorganic peroxygen compounds include alkali metal **perborate** and percarbonate materials, most preferably the percarbonates. For example, sodium **perborate** (e.g. mono- or tetra-hydrate) can be used. Suitable inorganic bleaching agents can also

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include sodium or potassium **carbonate** peroxyhydrate and equivalent "percarbonate" bleaches, sodium pyrophosphate peroxyhydrate, urea peroxyhydrate, and sodium **peroxide**. Persulfate bleach (e.g., OXONE, manufactured commercially by DuPont) can also be used. Frequently inorganic peroxygen bleaches will be coated with silicate, borate, sulfate or water-soluble surfactants. For example, coated percarbonate particles are available from various commercial sources such as FMC, Solvay Interox, Tokai Denka and Degussa.

Inorganic peroxybleach

----- 5521293
classes:1 536/172 1 536/53 1 536/54
score: 422

keywords: quaternary ammonium;hydrogen peroxide;peroxide;residue;dimethyl;glycol ether;amide;organic solvent;organic;perborate;hydrogen peroxide;phthalic;benzoic;aromatic;carboxylic acid;carboxylic;carbonate;hydroxide;quaternary ammonium;quaternary;peroxide;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;glycol;solvent;dropwise;heated;aluminum;washed;oxidized;sucrose;sugar;hydroxyl;formic;solvents;monobutyl ether;monobutyl;glycol monoethyl;monoethyl;glycol monomethyl;monomethyl;dipropylene;propylene;diethylene;ethylene glycol;amines;diamines;ethanolamine;monoethanolamine;benzyl;propionic acid;propionic;adipic;lactic;acetic acid;acetic;aliphatic;ammonium salts;alkyl;potassium;ammonia;crystallization;anhydride;alkaline;disolved;phase;insoluble;dioxide;alkali;acidic;dissolve;decomposition;inorganic;residue;dimethyl;glycol ether;amide;organic solvent;organic;perborate;hydrogen peroxide;phthalic;benzoic;aromatic;carboxylic acid;carboxylic;carbonate;hydroxide;quaternary ammonium;peroxide;Address;

- /C.sub.10 oxypropyl
D-gluconamide has a favorably small area per molecule value and is expected to pack tightly at the interface. The area per molecule (a.sub.m) of this compound was found to be similar to dodecyltri(oxyethylene) ether. This finding suggests that the heteroatom compounds of the invention to be surface-active and so these compounds are expected to deliver favorable surfactant benefits.

Example 91

Micellar Shape and Structure

The shape or type of micelle produced by a surfactant in aqueous solution is an important criteria for delivering certain surfactant benefits such as viscosity, detergency, foaming, wetting or emulsification. At present there appears to be four major types of micelles a surfactant can form in aqueous solution;

- (1) spherical micelles
- (2) cylindrical rod-like micelles
- (3) lamellar disk-like micelles
- (4) vesicles or reversed micelles

In aqueous media, surfactant molecules may be oriented in all these

----- 5710121
classes:1 510/467 1 510/495 1 510/497 1 510/506
score: 421

keywords: residue;organic;aromatic;carboxylic acid;carboxylic;carboxylate;hydroxide;ammonium;oxide;salt;acid;hydrogen;soluble;ether;glycol;solvent;disclosures;dropwise;washed;sucrose;phenol;hydroxyl;ethers;propylene glycol;propylene;ethanol;acetic;aliphatic;ammonium salts;alkyl;anhydride;alkaline;dissolved;phase;alkali;inorganic;stripped;residue;organic;aromatic;carboxylic acid;carboxylic;carboxylate;hydroxide;

- a small amount of monoethanolamine. The NMR agreed with the structure.

EXAMPLE V--PREPARATION OF PHOSPHATE ESTERS

Phosphate Esters salts were prepared by the following procedure:

Methylenebis(octylphenol) sodium phosphate: A solution of Triethylamine (1.43 g, 14.15 mmol), methylene bis(octylphenol) (3.0 g, 7.0 mmol) in dry hexane/THF was added **dropwise** to a solution of phosphorous oxychloride (1.30 mL, 14.15 mmol) in small amount of hexane under argon at -5.degree. C. A white precipitate appeared. After stirring 2.5 hours at 0.degree. C., TLC (CHCl₃ : CH₃OH=5:1) showed that all starting material disappeared, the reaction was stopped by filtered off triethylammonium chloride salt. The solvent in filtrate was evaporated under reduced pressure.

A solution of NaOH in ice water was added to the filtrate at 0.degree. C. Final pH of the solution was about 13. The solution was stirred for two

hours before it was extracted with n

----- 6423676

classes:1 510/313 1 252/18638 1 510/303 1 510/372 1 510/376 1 510/499 1 564/123 1 564/199
score: 418

keywords: peroxy;diamine;hydrogen peroxide;peroxide;residue;dimethyl;organic;percarbonate;perborate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;quaternary;peroxide;peracid;peroxyhydrate;lactam;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;disclosures;dropwise;alumnum;washed;additive;enzyme;sugar;phenol;ethylenediaminetetraacetic acid;ethylenediaminetetraacetic;solvents;amides;ethylenediamine;moiety;acetic;aliphatic;ammonium salts;alkalinity;aryl;alkyl;potassium;urea;anhydride;alkaline;species;alkali;acidic;inorganic;residue;dimethyl;organic;percarbonate;perborate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;quaternary;peroxide;

- ordinary skill in the art will recognize that the present invention is not limited to the specific examples herein described or the ingredients and steps contained therein, but rather, may be practiced according to the broader aspects of the disclosure.

EXAMPLE I

Preparation of N-Methoxy-N-nanonoyl Acetamide (2)

Synthesis of N-Methoxynonanamide (1):

#STR14##

A 100 mL three-neck round-bottom flask equipped with a mechanical stirrer, argon inlet and pressure equalizing addition funnel is charged with ethylacetate (40 mL) and 1 M aqueous solution of potassium **carbonate** (20 mL, 2 eq). The reaction mixture is cooled in a salt water/ice bath. Methoxyamine hydrochloride (5.06 g, 1 eq) is added to the reaction mixture followed by a **dropwise** addition of nonanoyl chloride (10.58 g, 1 eq). The reaction stirs at room temperature overnight. The reaction is diluted with ethylacetate and separated from the aqueous. The **organic** layer is **washed**

- try detergents are exemplified by the following formulations.

INGREDIENT	A	B	C	D	E
Bleach Activator*	%	%	%	%	%
Bleach Activator*	5	3.5	1	3.5	2
Sodium Percarbonate	0	0	19	21	0
Sodium **perborate** monohydrate	21	21	0	0	20
Conventional Bleach Activator	0	0	3	1	0
Linear alkylbenzenesulfonate	5.5	11	19	12	9.5
Alkyl ethoxylate (C45E7)	4	0	3	4	6
Zeolite A	20	20	9.5	17	21
SKS-6 .RTM. silicate (Hoechst)	0	0	11	11	0
Trisodium citrate	5	5	2	3	3
Acrylic Acid/Maleic Acid copolymer	4	0	4	5	0
Sodium polyacrylate	0	3	0	0	3
Chelant	0.4	0	0.4	0	0
Carboxymethylcellulose	0.3				
A	.10	.10	.10		.10
Bleach Activator.sup.2	3.5	3.5	2		7
Citric Acid	0.5	0	0		0.5
NaOH	to pH 4	to pH 4	to pH 4	to pH 4	
Hydrogen **peroxide**	6	0	0		7
Water	Balance	Balance	Balance	Balance	
	to 100%	to 100%	to 100%	to 100%	

.sup.1 Alkyl ethoxylate available from The Shell Oil Company.

.sup.2 Bleach Activator according to any of Examples I-VII.

The compositions are used as bleach boosting additive (to be used in ADDITION to a bleach OR non-bleach detergent such as TIDE.RTM.) in a wash test otherwise similar to that used in Example V. The additive is used at 1000 ppm, and the commercial detergent is used at 1000 ppm.

EXAMPLE X

A granular automatic dishwashing detergent composition comprises the following.

A

----- 6514925

classes:1 510/313 1 510/303 1 510/372 1 510/376 1 510/499 1 252/18638 1 560/42 1 564/123 1 564/154 1 564/199
155 1 564/199

score: 415

keywords: peroxy; diamine; hydrogen peroxide; peroxide; residue; dimethyl; organic; percarbonate; perborate; hydrogen peroxide; aromatic; carboxylic acid; carboxylic; carboxylate; carbonate; hydroxide; quaternary; peroxide; peracid; peroxyhydrate; lactam; ammonium; oxide; salt; acid; hydrogen; amine; soluble; ether; disclosures; dropwise; alumnum; washed; additive; enzyme; sugar; phenol; ethylenediaminetetraacetic acid; ethylenediaminetetraacetic; solvents; amides; ethylenediamine; moiety; acetic; aliphatic; ammonium salts; alkalinity; aryl; alkyl; potassium; urea; anhydride; alkaline; species; alkali; inorganic; residue; dimethyl; organic; percarbonate; perborate; hydrogen peroxide; aromatic; carboxylic acid; carboxylic; carboxylate; carbonate; hydroxide; quaternary; peroxide;

- prise most any fabric capable of being laundered in normal consumer use conditions. The laundry liquor includes the added bleach additive or bleaching composition containing a unsymmetrical acyclic imide activator as fully described above. The laundry liquor may also include any of the above described additives to the compositions such as **hydrogen **peroxide** source, detergents, chelates, and detergents enzymes. The compositions are preferably employed at concentrations of at least about 50 ppm and typically from about 1,000 to about 10,000 ppm in solution. The water temperatures preferably range from about 25.degree. C. to about 50.degree. C. The water to fabric ratio is preferably from about 1:1 to about 15:1.

Methods for washing soiled dishes such as tableware, also involve contacting the soiled dishes with an aqueous dishwashing liquor. The dishwashing liquor includes the added bleach additive or bleaching comp

- xynonanamide (1):
##STR14##

A 100 mL three-neck round-bottom flask equipped with a mechanical stirrer, argon inlet and pressure equalizing addition funnel is charged with ethylacetate (40 mL) and 1 M aqueous solution of potassium **carbonate** (20 mL, 2 eq). The reaction mixture is cooled in a salt water/ice bath. Methoxyamine hydrochloride (5.06 g, 1 eq) is added to the reaction mixture followed by a **dropwise** addition of nonanoyl chloride (10.58 g, 1 eq). The reaction stirs at room temperature overnight. The reaction is diluted with ethylacetate and separated from the aqueous. The **organic** layer is **washed** with deionized water (1.times.50 mL), dried over sodium sulfate, filtered, and concentrated under vacuum.

Synthesis of N-Methoxy-N-nonanoyl Acetamide (2):

##STR15##

A 100 mL round-bottom flask equipped with a magnetic stir bar, argon inlet and pressure equalizing addition funnel is charged with methylene chloride (22.5 mL),

----- 4689168
classes:1 510/417 1 510/182 1 510/432 1 510/466 1 510/505 1 510/506
score: 415

keywords: dimethyl; organic solvent; organic; carboxylate; ammonium hydroxide; hydroxide; ammonium; oxide; salt; acid; hydrogen; amine; soluble; ether; glycol; solvent; Tertiary; glycol monobutyl; monobutyl; glycol monoethyl; monoethyl; ethers; propylene; diethylene glycol; diethylene; ethylene glycol; glycols; ethylenediamine; ethanol; moiety; aliphatic; ammonium salts; alkalinity; aryl; alkyl; ammonia; phase; alkali; unstable; dimethyl; organic solvent; organic; carboxylate; ammonium hydroxide; hydroxide;

- lder salts, alkalinity agents, buffering agents, and the like. Typically, these adjuvants are present in amounts of from 0 to about 1.0% by weight, on an active constituent basis.

The dye and perfume are added to improve the aesthetics of the composition, while the builder salts and alkalinity agents are functional adjuvants in that the efficacy of the composition is improved by their inclusion. Certain builders are inherent alkalinity agents, the increase in the pH of the composition provided by the incorporation thereof improving the efficacy of the nonvolatile surfactant, as is known in the art. Other builders (or the same builders) may also provide chelating or sequestering properties, if desired. Still other builders may provide buffering properties.

The builders useful for incorporation in the composition include the ammonium and alkali metal **carbonate**, citrate, phosphate, and silicate salts, preferably the sodium salts

----- 6399557

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classes:1 510/310 1 252/18633 1 510/220 1 510/221 1 510/224 1 510/302 1 510/303 1 510/304 1 510/311 1 510/312 1 510/313 1 510/314 1 510/372 1 510/376
score: 394

keywords: peroxy; diamine; quaternary ammonium; hydrogen peroxide; peroxide; residue; dimethyl; amide; organic; percarbonate; perborate; persulfate; hydrogen peroxide; aromatic; carboxylic acid; carboxylic; carboxylate; carbonate; hydroxide; quaternary ammonium; quaternary; peroxide; peracid; hydroperoxide; peroxyhydrate; sulfoxide; lactam; ammonium; oxide; salt; acid; hydrogen; amine; soluble; ether; glycol; solvent; disclosures; distilled; heated; benzoate; aluminum; washed; additive; oxidized; metal salt; enzyme; sucrose; glucose; phenol; hydroxyl; solvents; ethers; propylene; diethylene glycol; diethylene; ethylene glycol; glycols; amides; amines; corrosion; ethylenediamine; ethanol; monoethanolamine; benzyl; moiety; adipic; acetic; aliphatic; ammonium salts; alkalinity; aryl; alkyl; peracetic acid; metal ions; potassium; sodium salt; ammonia; urea; anhydride; alkaline; oxides; dissolved; phase; species; insoluble; alkali; acidic; decomposition; inorganic; residue; dimethyl; amide; organic; percarbonate; perborate; persulfate; hydrogen peroxide; aromatic; carboxylic acid; carboxylic; carboxylate; carbonate; hydroxide; quaternary ammonium; quaternary; peroxide;

- duct is preferably predissolved in warm water before adding to the washing appliance if desired.

EXAMPLE 13

Dithiocyanato Manganese (II) 5,8
dimethyl-1,5,8,12-tetraazabicyclo[10.3.2]heptadecane Synthesis

##STR56##

Synthesis of 1,5,9,1 3-Tetraazatetracyclo[11.2.2.2.sup.5,9]heptadecane
1,4,8,12-tetraazacyclopentadecane (4.00 g, 18.7 mmol) is suspended in
acetonitrile (30 mL) under nitrogen and to this is added glyoxal (3.00 g,
40% aqueous, 20.7 mmol). The resulting mixture is **heated** at 65.degree. C.
for 2 hours. The acetonitrile is removed under reduced pressure. **distilled**
water (5 mL) is added and the product is extracted with chloroform
(5.times.40 mL). After drying over anhydrous sodium sulfate and
filtration, the solvent is removed under reduced pressure. The product is
then chromatographed on neutral alumina (15.times.2.5 cm) using
chloroform/methanol (97.5:2.5 increasing to 95:5). The solvent is removed
under red

----- 6387862
classes:1 510/311 1 510/376 1 510/500
score: 386

keywords: peroxy; diamine; quaternary ammonium; hydrogen peroxide; peroxide; residue; dimethyl; amide; organic; percarbonate; perborate; persulfate; hydrogen peroxide; aromatic; carboxylic acid; carboxylic; carboxylate; carbonate; hydroxide; quaternary ammonium; quaternary; peroxide; peracid; hydroperoxide; peroxyhydrate; sulfoxide; lactam; ammonium; oxide; salt; acid; hydrogen; amine; soluble; ether; glycol; solvent; disclosures; distilled; heated; benzoate; aluminum; washed; additive; oxidized; metal salt; enzyme; sucrose; glucose; phenol; hydroxyl; solvents; ethers; propylene; diethylene glycol; diethylene; ethylene glycol; glycols; amides; amines; corrosion; ethylenediamine; ethanol; monoethanolamine; benzyl; moiety; adipic; acetic; aliphatic; ammonium salts; alkalinity; aryl; alkyl; peracetic acid; metal ions; potassium; sodium salt; ammonia; urea; anhydride; alkaline; oxides; dissolved; phase; species; insoluble; alkali; acidic; decomposition; inorganic; residue; dimethyl; amide; organic; percarbonate; perborate; persulfate; hydrogen peroxide; aromatic; carboxylic acid; carboxylic; carboxylate; carbonate; hydroxide; quaternary ammonium; quaternary; peroxide;

- hon CG, sold by Rohm & Has
.sup.5 Copolymer of propylene terephthalate and ethyleneoxide
.sup.6 Mn(Bcyclam)Cl.sub.2 as in Synthesis Example 1

EXAMPLE 27

Dithiocyanato Manganese (II)
5,8 **dimethyl**-1,5,8,12-tetraazabicyclo[10.3.2]heptadecane Synthesis

##STR54##

Synthesis of 1,5,9,13-Tetraazatetracyclo[11.2.2.2.sup.5,9]heptadecane
1,4,8,12-tetraazacyclopentadecane (4.00 g, 18.7 mmol) is suspended in
acetonitrile (30 mL) under nitrogen and to this is added glyoxal (3.00 g,
40% aqueous, 20.7 mmol). The resulting mixture is **heated** at 65.degree. C.
for 2 hours. The acetonitrile is removed under reduced pressure. **distilled**
water (5 mL) is added and the product is extracted with chloroform
(5.times.40 mL). After drying over anhydrous sodium sulfate and
filtration, the solvent is removed under reduced pressure. The product is
then chromatographed on neutral alumina (15.times.2.5 cm) using
chloroform/methanol (97.5:2.5 increasing to 95:5). The solv

----- 4371517

classes:1 424/7013 1 8/1157 1 8/1275 1 8/12751 1 424/7011 1 424/7016 1 424/7017 1 424/7031
score: 381

keywords: diamine;quaternary ammonium;organic solvent;organic;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;ammonium salt;quaternary ammonium;quaternary;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;glycol;solvent;heated;Tertiary;propionate;washed;immersed;metal salt;sucrose;hydroxyl;organic solvents;solvents;monobutyl;monoethyl ether;glycol monoethyl;monoethyl;monomethyl ether;glycol monomethyl;monomethyl;propylene;diethylene glycol;diethylene;ethylene glycol;benzyl;adipic acid;adipic;aliphatic;ammonium salts;alkyl;potassium;sodium salt;ammonia;anhydride;alkali;inorganic;organic solvent;organic;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;ammonium salt;quaternary ammonium;quaternary;

- occurring units of the formula

##STR19##

PAQ-3: Polymer having recurring units of the formula

##STR20##

n being equal to about 6. GAFQUAT 755: **quaternary** vinylpyrrolidone copolymer having a molecular weight of 1,000,000, marketed by GENERAL ANILINE.

MERQUAT 550: Dimethyldiallylammonium chloride/acrylamide copolymer having a molecular weight >500,000, sold by MERCK.

PD 170: Adipic acid/epoxypropyl-diethylenetriamine copolymer sold by HERCULES, of the formula

##STR21##

POLYQUART H: Polyglycol-polyamine polycondensate sold by HENKEL.

CARTARETINE F4: Adipic acid/dimethylaminohydroxypropyl diethylenetriamine copolymer sold by SANDOZ.

MERQUAT 100: Dimethyldialyl ammonium homopolymer of molecular weight below 100,000 sold by MERCK.

POLYMIN H.S.: Polyethyleneimine of d.sub.20 =about 1.07 and viscosity 500-1,000 cps as a 20% aqueous solution sold by BASF.

SURFACE ACTIVE AGENTS

TA-1:

##STR22##

R: C.sub.9 -C.sub.12

----- 6093343

classes:1 252/18633 1 510/311 1 510/349 1 510/376 1 510/441 1 510/445 1 510/446 1 510/508
score: 380

keywords: hydrogen peroxide;peroxide;residue;organic;percarbonate;perborate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;quaternary;peroxide;peracid;ammonium;oxide;salt;acid;hydrogen;amine;soluble;glycol;solvent;distilled;benzoate;aluminum;oxidized;enzyme;sucrose;glucose;sugar;phenol;ethylenediaminetetraacetic;propylene glycol;propylene;diethylene;glycols;amines;corrosion;ethylenediamine;monoethanolamine;moiety;acetic;aliphatic carboxylic;aliphatic;ammonium salts;aryl/alkyl;potassium;ammonia;urea;alkaline;oxides;dissolved;species;alkali;inorganic;residue;organic;percarbonate;perborate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;quaternary;peroxide;Address;

- 951 A to N ovo. See also WO 920524 9 and RD 94359044.

Cutinase enzymes suitable for use herein are described in WO 8809367 A to Genencor.

Peroxidase enzymes may be used in combination with oxygen sources, e.g.,

percarbonate, **perborate**, hydrogen **peroxide**, etc., for "solution bleaching" or prevention of transfer of dyes or pigments removed from substrates during the wash to other substrates present in the wash solution. Known peroxidases include horseradish peroxidase, ligninase, and haloperoxidases such as chloro- or bromo-peroxidase. Peroxidase-containing detergent compositions are disclosed in WO 8909813 A, Oct. 19, 1989 to Novo and WO 8909813 A to Novo.

A range of enzyme materials and means for their incorporation into synthetic detergent compositions is also disclosed in WO 9307263 A and WO 9307260 A to Genencor International, WO 8908694 A to Novo, and U.S. Pat. No. 3,553,139, Jan. 5, 1971 to McCarty et al. Enzymes are fur

- ut 8%, most preferably from about 0.01% to about 6%, by weight of an enzyme stabilizing system. The enzyme stabilizing system can be any stabilizing system which is compatible with the detergative enzyme. Such a system may be inherently provided by other formulation actives, or be added separately, e.g., by the formulator or by a manufacturer of detergent-ready enzymes. Such stabilizing systems can,

for example, comprise calcium ion, boric acid, propylene glycol, short chain **carboxylic acid**'s, boronic acids, and mixtures thereof, and are designed to address different stabilization problems depending on the type of enzyme and type of detergent composition.

One stabilizing approach is the use of water-soluble sources of calcium and/or magnesium ions in the composite particles or in the finished compositions which provide such ions to the enzymes. Calcium ions are generally more effective than magnesium ions and are preferred herei - sphate, condensed phosphate, acetate, **benzoate**, citrate, formate, **lactate**, malate, tartrate, **salicylate**, etc., and mixtures thereof can be used if desired. In general, since the chlorine scavenger function can be performed by ingredients separately listed under better recognized functions, (e.g., hydrogen **peroxide** sources), there is no absolute requirement to add a separate chlorine scavenger unless a compound performing that function to the desired extent is absent from an enzyme-containing embodiment of the invention; even then, the scavenger is added only for optimum results. Moreover, the formulator will exercise a chemist's normal skill in avoiding the use of any enzyme scavenger or stabilizer which is majorly incompatible, as formulated, with other reactive ingredients, if used. In relation to the use of **ammonium salt**'s, such salts can be simply admixed with the detergent composition but are prone to adsorb water and

6369024
classes:1 510/499 1 510/350 1 510/503 1 510/504
score: 379

keywords: diamine;tetramethyl;dimethyl;amide;organic;percarbonate;perborate;persulfate;carboxylic;carbonate;peroxyhydrate;ammonium;oxide;salt;acid;amine;ether;solvent;disclosures;heated;additive;enzyme;glucose;sugar;phenol;ethers;propylene;amides;amines;ethylenediamine;ethanolamine;ethanol;ammonium salts;alkalinit;y;alkyl;potassium;urea;dissolved;alkali;inorganic;stripped;dimethyl;amide;organic;percarbonate;perborate;persulfate;carboxylic;carbonate;Address;

- compositions are prepared containing various linear amine based polymer, oligomer or copolymer materials. Such granular detergent compositions all have the following formulas:

Component	Example 1	Example 2
	Wt %	Wt %
C.sub.12 Linear alkyl benzene sulfonate	9.40	9.40
C.sub.14-15 alkyl sulfonate	11.26	11.26
Zeolite Builder	27.79	27.79
Sodium **carbonate**	27.31	27.31
PEG 4000	1.60	1.60
Dispersant	2.26	2.26
C.sub.12-13 alkyl ethoxylate (E9)	1.5	1.5
Sodium **perborate**	1.03	1.03
Soil Release Polymer	0.41	0.41
Enzymes	0.46	0.46
Condensate of bis(hexamethylene)triamine and epichloro	0.8	1.6
- Moisture, Sulfate		100%

TABLE II

#	Polymer*	wt. %
3	Bis(hexamethylene)triamine	1.6
4	Bis(hexamethylene)triamine	3.2
5	Adduct of Aminopropyldiethanolamine and epi (ratio 2:1)	0.8
6	Adduct of N,N bis(3-aminopropyl)methylamine and epi (Ratio 2:1)	0.8
7	Adduct of N-(2-Aminoethyl) ethanolamine and epi (Ratio 2:1)	0.8
8	Adduct of N,N bis(aminopropyl)ethylenediamine and epi (Ratio 2:1)	0.8
9	Dipropylenetriamine-epi 2:1	0.8
10	Adduct of 2 amino-5 (diethylamino) pentane and epi (ratio 2:1)	0.8
11	Adduct of Diethylaminoethylamine and epi (Ratio 2:1)	0.8
12	Adduct of 2(ethylamino) ethyleneamine and epi (Ratio 1.5:1)	0.8
13	Adduct of 4,9 Dioxadodecane, 1,12 **diamine** and epi (Ratio 2:1)	0.8
14	Adduct of 4,7,10 trioxitri	

- mer, oligomer or copolymer materials. Such liquid detergent compositions all the following formulas:

Component	Examples 24-31 Wt. %
C.sub.12-15 alkyl ether (2.5) sulfate	19.0
C.sub.12-13 alkyl ethoxylate (9.0)	2.00
C.sub.12-14 glucose **amide**	3.50
Citric Acid	3.00
C.sub.12-14 Fatty Acid	2.00
MEA	to pH 8
Ethanol	3.4
Propanediol	6.51
Borax	2.5
Dispersant	1.18
Na Toluene Sulfonate	2.50
Oligomer and/or polymer as shown in Table III as in Table III	
Dye, Perfume, Brighteners, Enzymes, Preservatives,	

----- 6395701
 classes:1 510/437 1 510/481 1 554/1
 score: 378

keywords: peroxy; diamine; quaternary ammonium; hydrogen peroxide; peroxide; residue; titanium; dimethyl; glycol ether; amide; organic; percarbonate; perborate; hydrogen peroxide; salicylic; benzoic; aromatic; carboxylic acid; carboxylic; carboxylate; carbonate; quaternary ammonium; quaternary; peroxide; hydroperoxide; peroxyhydrate; lactam; ammonium; oxide; salt; acid; hydrogen; amine; soluble; ether; glycol; solvent; disclosures; distilled; heated; lactate; benzoate; Severe; aluminum; copper; washed; additive; enzyme; sucrose; glucose; sugar; phenol; solvents; monomethyl ethers; dipropylene; propylene glycol; propylene; diethylene; amides; amines; corrosion; ethylenediamine; ethanol; diethanolamine; benzyl; moiety; adipic; lactic acid; lactic acid; acetic; aliphatic; ammonium salts; alkalinity; aryl; alkyl; potassium; sodium salt; ammonia; anhydride; alkaline; oxides; dissolved; phase; species; insoluble; titanium dioxide; dioxide; alkali; acidic; decomposition; inorganic; residue; titanium; dimethyl; glycol ether; amide; organic; percarbonate; perborate; hydrogen peroxide; salicylic; benzoic; aromatic; carboxylic acid; carboxylic; carboxylate; carbonate; quaternary ammonium; quaternary; peroxide;

- 0.30 0.30 0.30 0.30 0.30 0.30
 distilled Water . Bal. Bal. Bal. Bal. Bal. Bal.
 DEQA2 is Di(acyloxyethyl) (2-hydroxyethyl)methyl ammonium methyl sulfate where the acyl group is derived from a partially hydrogenated canola fatty acid 85% active.
 DEQA8 is Di(acyloxyethyl) (2-hydroxyethyl)methyl ammonium methyl sulfate where the acyl group is derived from a mixture of partially hydrogenated canola fatty acid and a mixture of the branched acids of examples 16-23, 85% active.
 DEQA10 is Di(acyloxyethyl) (2-hydroxyethyl)methyl ammonium methyl sulfate where the acyl group is derived from a mixture of partially hydrogenated canola fatty acid and a mixture of the branched acids of examples 24 to 30, 85% active.
 DEQA24 is Di(acyloxyethyl) (2-hydroxyethyl)methyl ammonium methyl sulfate where the acyl group is derived from a mixture of partially hydrogenated canola fatty acid and the branched acid of ex

----- 6239093
 classes:1 510/352 1 510/119 1 510/351 1 510/428 1 510/492
 score: 378

keywords: quaternary ammonium; hydrogen peroxide; peroxide; residue; dimethyl; glycol ether; amide; organic solvent; organic; perborate; persulfate; hydrogen peroxide; benzoic acid; benzoic; aromatic; carboxylic acid; carboxylic; carbonate; hydroxide; quaternary ammonium; quaternary; peroxide; peroxyhydrate; sulfone; ammonium; oxide; salt; acid; hydrogen; amine; soluble; ether; glycol; solvent; disclosures; distilled; heated; Tertiary; copper; washed; additive; enzyme; sucrose; glucose; sugar; phenol; hydroxyl; solvents; monobutyl ether; glycol monobutyl; monobutyl; glycol monoethyl; monoethyl; ethers; dipropylene glycol; dipropylene; propylene glycol; propylene; triethylene glycol; triethylene; diethylene glycol; diethylene; ethylene glycol; glycols; amides; amines; corrosion; ethylenediamine; ethanol; triethanolamine; diethanolamine; monoethanolamine; benzyl; moiety; propionic acid; propionic; aliphatic; ammonium salts; alkalinity; aryl; alkyl; potassium; urea; anhydride; alkaline; oxides; dissolved; phase; species; insoluble; dioxide; alkali; dissolve; inorganic; exposure; residue; dimethyl; glycol ether; amide; organic solvent; organic; perborate; persulfate; hydrogen peroxide; benzoic acid; benzoic; aromatic; carboxylic acid; carboxylic; carbonate; hydroxide; quaternary ammonium; quaternary; peroxide;

- allyl ether groups per polyol molecule. When the

polyol is sucrose, it is preferred that the sucrose have at least about five allyl ether groups per sucrose molecule. It is preferred that the polyether of the polyol comprise from about 0.1% to about 4% of the total monomers, more preferably from about 0.2% to about 2.5%.

Preferred monomeric olefinically unsaturated **carboxylic acid**s for use in producing carboxyvinyl polymers used herein include monomeric, polymerizable, alpha-beta monoolefinically unsaturated lower aliphatic **carboxylic acid**s; more preferred are monomeric monoolefinic acrylic acids of the structure

##STR30##

where R is a substituent selected from the group consisting of hydrogen and lower alkyl groups; most preferred is acrylic acid.

Preferred carboxyvinyl polymers have a molecular weight of at least about 750,000; more preferred are carboxyvinyl polymers having a molecular weight of at least about 1,250,000; mo

- monomer units and other non-cationic units referred to herein as spacer monomer units. Such polymers are known in the art, and a variety can be found in International Cosmetic Ingredient Dictionary, Fifth Edition, 1993, which is incorporated by reference herein in its entirety.

Suitable optional cationic polymers include, for example, copolymers of vinyl monomers having cationic amine or **quaternary ammonium** functionalities with water soluble spacer monomers such as acrylamide, methacrylamide, alkyl and dialkyl acrylamides, alkyl and dialkyl methacrylamides, alkyl acrylate, alkyl methacrylate, vinyl caprolactone, and vinyl pyrrolidone. The alkyl and dialkyl substituted monomers preferably have C.sub.1 -C.sub.7 alkyl groups, more preferably C.sub.1 -C.sub.3 alkyl groups. Other suitable spacer monomers include vinyl esters, vinyl alcohol (made by hydrolysis of polyvinyl acetate), maleic anhydride, propylene glycol, and ethylene g

----- 6228829

classes:1 510/357 1 510/424 1 510/426 1 510/428 1 560/76 1 568/458 1 568/882
score: 372

keywords: peroxy; diamine; quaternary ammonium; peroxide; residue; dimethyl sulfoxide; dimethyl; amide; organic; p ercarbonate; perborate; persulfate; aromatic; carboxylic acid; carboxylate; carbonate; hydroxide; quaternary ammonium; quaternary; peroxide; peroxyhydrate; sulfoxide; lactam; ammonium; oxide; salt; acid; hydrogen; amine; soluble; ether; glycol; solvent; disclosures; heated; aluminum; washed; additive; metal salt; enzyme; glucose; sugar; phenol; hydroxyl; ethylenediaminetetraacetic; propylene glycol; propylene; diethylene; glycals; amides; amines; ethylenediamine; ethanol; triethanolamine; monoethanolamine; benzyl; moiety; acetic; aliphatic; ammonium salts; alkyl; potassium; ammonia; crystallization; urea; anhydride; alkaline; oxides; dissolved; phase; species; insoluble; alkali; acidic; inorganic; residue; dimethyl sulfoxide; dimethyl; amide; organic; percarbonate; perborate; persulfate; aromatic; carboxylic acid; carboxylic; carboxylate; carbonate; hydroxide; quaternary ammonium; quaternary; peroxide;

- repared in accord with the invention:

	L	M	N	O	P
MBAS14.4	16.5	12.5	8.5	4	1-25
Any Combination of:	0-6	10	14	18.5	0-20
C45 AS					
C45E1S					
LAS					
C16 SAS					
C14-17 NaPS					
C14-18 MES					
MBAE2S14.3					
AOA	0-2	0-2	0-2	0-2	0-4
TFAA	1.6	1.6	1.6	1.6	0-4
C24E3, C23E6.5 or	5	5	5	5	0-6
MBAE14					
Zeolite A	15	15	15	15	10-30
NaSKS-6	11	11	11	11	5-15
Citrate	3	3	3	3	0-8
MA/AA	4.8	4.8	4.8	4.8	0-8
HEDP	0.5	0.5	0.5	0.5	0-1
carbonate	8.5	8.5	8.5	8.5	0-15
Percarbonate or PB1	20.7	20.7	20.7	20.7	0-25
TAED	4.8	4.8	4.8	4.8	0-

----- 4597898

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classes:1 510/325 1 510/321 1 510/340 1 510/341 1 510/352 1 510/423 1 510/425 1 510/499
score: 369

keywords: diamine;quaternary ammonium;dimethyl;amide;organic;carboxylic;carboxylate;carbonate;hydroxide;q uaternary ammonium;quaternary;ammonium;oxide;acid;hydrogen;amine;ether;glycol;Tertiary;washed;enzyme;gluc ose;phenol;ethers;propylene;triethylene;diethylene;amides;amines;corrosion;diamines;ethylenediamine;ethan ol;triethanolamine;monoethanolamine;benzyl;moiety;acetic;aliphatic carboxylic;aliphatic;ammonium salts;ar yl;alkyl;potassium;ammonia;anhydride;alkaline;oxides;phase;solubilized;alkali;inorganic;dimethyl;amide;or ganic;carboxylic;carboxylate;carbonate;hydroxide;quaternary ammonium;quaternary;

-
Highly preferred polycarboxylate builders are disclosed in U.S. Pat. No. 3,308,067 to Diehl, issued Mar. 7, 1967 (herein incorporated by reference). Such materials include the water-soluble salts of homo- and copolymers of aliphatic **carboxylic acid**'s such as maleic acid, itaconic acid, mesaconic acid, fumaric acid, aconitic acid, citraconic acid and methylenemalic acid.

Other builders include the **carboxylate**'d carbohydrates disclosed in U.S. Pat. No. 3,723,322 to Diehl issued Mar. 28, 1973 (herein incorporated by reference).

Other useful builders are sodium and potassium carboxymethyloxymalonate, carboxymethyloxysuccinate, cis-cyclohexanehexacarboxylate, cis-cyclopentanetetracarboxylate phloroglucinol trisulfonate, water-soluble polyacrylates (having molecular weights of from about 2,000 to about 200,000 for example), and the copolymers of maleic anhydride with vinyl methyl ether or ethylene.

Other suitable polycarboxylates a

- micron particle diameter; 2.7:1 wt. mixtures of sodium sesquicarbonate and calcium **carbonate** having a particle diameter of 0.5 microns; 20:1 wt. mixtures of sodium sesquicarbonate and calcium **hydroxide** having a particle diameter of 0.01 micron; and a 3:3:1 wt. mixture of sodium **carbonate**, sodium aluminate and calcium oxide having a particle diameter of 5 microns.

OTHER OPTIONAL DETERGENT INGREDIENTS

Other optional ingredients which can be included in detergent compositions of the present invention, in their conventional art-established levels for use (i.e., from 0 to about 20%), include solvents, bleaching agents, bleach activators, soil-suspending agents, corrosion inhibitors, dyes, fillers, optical brighteners, germicides, pH adjusting agents (monoethanolamine, sodium **carbonate**, sodium **hydroxide**, etc.), enzymes, enzyme-stabilizing agents, perfumes, fabric softening components, static control agents, and the like.

DETERGENT FO

- degree. F.).

Built liquid detergent compositions can be in the form of single phase liquids provided that the builder is solubilized in the mixture at its level of use. Such liquids conventionally contain 10 to 25% total surfactant, 10 to 25% builder which can be **organic** or inorganic, 3 to 10% of a hydrotrope system and 40 to 77% water. Liquids of this type also have a low viscosity (100 to 150 centipoise at 75.degree. F.). Built liquid detergents incorporating components that form heterogeneous mixtures (or levels of builder that cannot be completely dissolved) can also comprise detergent compositions of the present invention. Such liquids conventionally employ viscosity modifiers to produce systems having plastic shear characteristics to maintain stable dispersions and to prevent phase separation or solid settlement.

NEAR NEUTRAL WASH pH DETERGENT FORMULATIONS

While the detergent compositions of the present invention are operativ

- of a cosurfactant selected from the group consisting of:

(i) **quaternary ammonium** surfactants having the formula:

[R.sup.2 (OR.sup.3).sub.y] [R.sup.4 (OR.sup.3).sub.y].sub.2 R.sup.5
N.sup.+ X.sup.-

wherein R.sup.2, each R.sup.3, R.sup.4, R.sup.5, X and y are as previously defined;

(ii) diquaternary ammonium surfactants having the formula:

[R.sup.2 (OR.sup.3).sub.y] [R.sup.4 (OR.sup.3).sub.y].sub.2 N.sup.+
R.sup.3 N.sup.+ R.sup.5 [R.sup.4 (OR.sup.3).sub.y].sub.2 (X.sup.-).sub.2

wherein R.sup.2, R.sup.3, R.sup.4, y and X are as defined above;
particularly preferred are the C.sub.8 -C.sub.16 alkyl pentamethylethylenediamine chloride, bromide and methylsulfate salts;

(iii) amine surfactants having the formula:

[R.sup.2 (OR.sup.3).sub.y] [R.sup.4 (OR.sup.3).sub.y].sub.2 N

wherein R.sup.2, R.sup.3, R.sup.4, R.sup.5 and y are as defined above;

particularly preferred are the C.sub.12 -C.sub.16 alkyl **dimethyl** amines;
 (iv) **diamine** surfactants having the formula:

- AMINES

A. Experimental Method

1. Clay Soil Removal

Clay soil removal comparisons were conducted in a standard 1 liter

Tergotometer employing water of 7 grain hardness (3:1 Ca.sup.++ :Mg.sup.++) and a temperature of 100.degree. F. Soiled swatches were **washed** in the Tergotometer for 10 minutes and rinsed twice with water (7 grain hardness) at 70.degree. F. for 2 minutes.

65% polyester/35% cotton blend fabric was used for the swatches. The swatches were 5 inches by 5 inches in size and were soiled by dipping in an aqueous slurry of local clay and subsequent baking to remove the water.

The dipping and baking was repeated 5 times.

One wash employed 2000 ppm of a control liquid detergent composition containing the following surfactants:

Surfactant	Amount (%)
Sodium C.sub.14 -C.sub.15 alkyl	
	10.8
ethoxysulfate	
C.sub.13 li	

----- 6087309
 classes:1 510/125 1 510/357 1 510/424 1 510/428 1 510/506 1 560/76 1 568/458 1 568/882
 score: 364

keywords: quaternary ammonium;hydrogen peroxide;peroxide;residue;dimethyl sulfoxide;dimethyl;glycol ether;amide;organic solvent;organic;perborate;persulfate;hydrogen peroxide;benzoic acid;benzoic;aromatic;carboxylic;carbonate;hydroxide;quaternary ammonium;quaternary;peroxide;peroxyhydrate;sulfoxide;sulfone;ammonium oxide;salt;acid;hydrogen;amine;soluble;ether;glycol;solvent;disclosures;distilled;heated;Tertiary;washe d;additive;enzyme;sucrose;glucose;sugar;phenol;hydroxyl;solvents;monobutyl ether;glycol monobutyl;monobut yl;glycol monoethyl;monoethyl;ethers;dipropylene glycol;dipropylene;propylene glycol;propylene;triethylen e glycol;triethylene;diethylene glycol;diethylene;ethylene glycol;glycols;amides;amines;corrosion;ethylen ediamine;ethanol;triethanolamine;diethanolamine;monoethanolamine;benzyl;moiety;propionic acid;propionic;a cetic;aliphatic;ammonium salts;alkalinity;aryl;alkyl;potassium;crystallization;urea;anhydride;alkaline;ox ides;dissolved;phase;species;insoluble;alkali;acidic;dissolve;inorganic;exposur e;residue;dimethyl sulfoxide;dimethyl;glycol ether;amide;organic solvent;organic;perborate;persulfate;hydrogen peroxide;benzoic acid;benzoic;aromatic;carboxylic;carbonate;hydroxide;quaternary ammonium;quaternary;peroxide;

- The cationic amines can be primary, secondary, or **Tertiary** amines, depending upon the particular species and the pH of the composition. In general, secondary and **Tertiary** amines, especially **Tertiary** amines, are preferred. The cationic polymers can comprise mixtures of monomer units derived from amine- and/or **quaternary ammonium**-substituted monomer and/or compatible spacer monomers.

Other cationic polymers that can be used include polysaccharide polymers, such as cationic cellulose derivatives, cationic starch derivative, and cationic guar gum derivatives. Other materials include **quaternary** nitrogen-containing cellulose ethers as described in U.S. Pat. No. 3,962,418, and copolymers of etherified cellulose and starch as described in U.S. Pat. No. 3,958,581, which descriptions are incorporated herein by reference.

The shampoo compositions of the present invention may comprise one or more optional ingredients to improve or otherwise

- materials.

Optional materials include foam boosters, preservatives, thickeners, cosurfactants, dyes, perfumes, solvents, styling polymers, anti-static agents, anti-dandruff aids, and pediculocides.

Preferred optional materials include foam boosters, especially fatty ester (e.g. C8-C22) mono- and di (C1-C5, especially C1-C3) alkanol amides, specific examples of which include coconut monoethanolamide and coconut diethanolamide.

Examples of other suitable optional materials include preservatives such as benzyl alcohol, methyl paraben, propyl paraben and imidazolidinyl urea; fatty alcohols; block polymers of ethylene oxide and propylene oxide such as Pluronic F88 offered by BASF Wyandotte; sodium chloride, sodium sulfate; ammonium xylene sulfonate; propylene glycol; polyvinyl alcohol; ethyl alcohol; pH adjusting agents such as citric acid, succinic acid, phosphoric acid, sodium **hydroxide**, sodium **carbonate**, etc.; perfumes; and

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dye
- mix tank and **heated** to about 74.degree. C. with slow agitation to form a surfactant solution. Cocamide monoethanolamide and fatty alcohol, as applicable, are added to the tank and allowed to disperse. Ethylene glycol distearate (EGDS) is then added to the mixing vessel, and melted. After the EGDS is well dispersed (usually after about 5 to 20 minutes) optional preservative are added and mixed into the surfactant solution. This mixture is passed through a heat exchanger where it is cooled to about 35.degree. C. and collected in a finishing tank. As a result of this cooling step, the ethylene glycol distearate crystallizes to form a crystalline network in the product. The remainder of the ammonium laureth sulfate, lauryl sulfate and other ingredients including the silicone premix are added to the finishing tank with ample agitation to insure a homogeneous mixture. A sufficient amount of the silicone premix is added to provi

----- 6376450
classes:1 510/392 1 435/221 1 510/300 1 510/305
score: 362

keywords: diamine;quaternary ammonium;hydrogen peroxide;peroxide;residue;titanium;dimethyl;amide;organic;percarbonate;perborate;persulfate;hydrogen peroxide;benzoic acid;benzoic;carboxylic;carbonate;hydroxide;quaternary ammonium;quaternary;peroxide;peracid;peroxyhydrate;lactam;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;glycol;solvent;Tertiary;benzoate;aluminum;washed;additive;enzyme;glucose;sugar;phenol;ethylene;diaminetetraacetic;solvents;monobutyl;glycol ethers;ethers;propylene glycol;propylene;diethylene;amides;amines;corrosion;ethylenediamine;ethanol;monoethanolamine;benzyl;moiety;aliphatic;ammonium salts;alkalinity;alkyl;metal ions;potassium;sodium salt;urea;alkaline;oxides;phase;species;insoluble;titanium dioxide;dioxide;solubilized;alkali;acidic;inorganic;residue;titanium;dimethyl;amide;organic;percarbonate;perborate;persulfate;hydrogen peroxide;benzoic acid;benzoic;carboxylic;carbonate;hydroxide;quaternary ammonium;quaternary;peroxide;

- Potassium **persulfate** 15.0 15.0 15.00 15.00
Sodium acid pyrophosphate 7.0 7.0 7.00 7.00
Pyrogenic silica 2.0 2.0 2.00 2.00
Tetracytylethylene diamine 7.0 7.0 7.00 7.00
Ricinoleylsulfosuccinate 0.5 0.5 0.50 0.50
Flavor 1.0 1.0 1.00 1.00

Alkaline Layer
Sodium **perborate** monohydrate 32.0 32.0 32.00 32.00
Sodium bicarbonate 19.0 19.0 19.00 19.00
EDTA 3.0 3.0 3.00 3.00
Sodium tripolyphosphate 12.0 12.0 12.00 12.00
PEG 20,000 2.0 2.0 2.00 2.00
Potassium **persulfate** 26.0 26.0 26.00 26.00
Sodium **carbonate** 2.0 2.0 2.00 2.00
Pyrogenic silica 2.0 2.0 2.00 2.00
Dye/flavor 2.0 2.0 2.00 2.00

Whil

----- 6046152
classes:1 510/428 1 510/119 1 510/122 1 510/125 1 510/426 1 510/427 1 510/506
score: 362

keywords: quaternary ammonium;hydrogen peroxide;peroxide;residue;dimethyl sulfoxide;dimethyl;glycol ether;amide;organic solvent;organic;perborate;persulfate;hydrogen peroxide;benzoic acid;benzoic;aromatic;carboxylic;carbonate;hydroxide;quaternary ammonium;quaternary;peroxide;peroxyhydrate;sulfoxide;sulfone;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;glycol;solvent;disclosures;distilled;heated;Tertiary;washed;additive;enzyme;sucrose;glucose;sugar;phenol;hydroxyl;solvents;monobutyl ether;glycol monobutyl;monobutyl;glycol monoethyl;monoethyl;ethers;dipropylene glycol;dipropylene;propylene glycol;propylene;triethylene glycol;triethylene;diethylene glycol;diethylene;ethylene glycol;glycols;amides;amines;corrosion;ethylenediamine;ethanol;triethanolamine;diethanolamine;monoethanolamine;benzyl;moyety;propionic acid;propionic;acetic;aliphatic;ammonium salts;alkalinity;aryl;alkyl;potassium;crystallization;urea;anhydride;alkaline;oxides;dissolved;phase;species;insoluble;alkali;acidic;dissolve;inorganic;exposure;residue;dimethyl sulfoxide;dimethyl;glycol ether;amide;organic solvent;organic;perborate;persulfate;hydrogen peroxide;benzoic acid;benzoic;aromatic;carboxylic;carbonate;hydroxide;quaternary ammonium;quaternary;peroxide;

- ene glycol.

The cationic amines can be primary, secondary, or **Tertiary** amines, depending upon the particular species and the pH of the composition. In

general, secondary and **Tertiary** amines, especially **Tertiary** amines, are preferred. The cationic polymers can comprise mixtures of monomer units derived from amine- and/or **quaternary ammonium**-substituted monomer and/or compatible spacer monomers.

Other cationic polymers that can be used include polysaccharide polymers, such as cationic cellulose derivatives, cationic starch derivative, and cationic guar gum derivatives. Other materials include **quaternary** nitrogen-containing cellulose ethers as described in U.S. Pat. No. 3,962,418, and copolymers of etherified cellulose and starch as described in U.S. Pat. No. 3,958,581, which descriptions are incorporated herein by reference.

The shampoo compositions of the present invention may comprise one or more optional ingredients to improve

-
y free of such materials.

Optional materials include foam boosters, preservatives, thickeners, cosurfactants, dyes, perfumes, solvents, styling polymers, anti-static agents, anti-dandruff aids, and pediculocides.

Preferred optional materials include foam boosters, especially fatty ester (e.g. C8-C22) mono- and di (C1-C5, especially C1-C3) alkanol amides, specific examples of which include coconut monoethanolamide and coconut diethanolamide.

Examples of other suitable optional materials include preservatives such as benzyl alcohol, methyl paraben, propyl paraben and imidazolidinyl urea; fatty alcohols; block polymers of ethylene oxide and propylene oxide such as Pluronic F88 offered by BASF Wyandotte; sodium chloride, sodium sulfate; ammonium xylene sulfonate; propylene glycol; polyvinyl alcohol; ethyl alcohol; pH adjusting agents such as citric acid, succinic acid, phosphoric acid, sodium **hydroxide**, sodium **carbonate**, etc.; perfumes - jacketed mix tank and **heated** to about 74.degree. C. with slow agitation to form a surfactant solution. Cocamide monoethanolamide and fatty alcohol, as applicable, are added to the tank and allowed to disperse. Ethylene glycol distearate (EGDS) is then added to the mixing vessel, and melted. After the EGDS is well dispersed (usually after about 5 to 20 minutes) optional preservative are added and mixed into the surfactant solution. This mixture is passed through a heat exchanger where it is cooled to about 35.degree. C. and collected in a finishing tank. As a result of this cooling step, the ethylene glycol distearate crystallizes to form a crystalline network in the product. The remainder of the ammonium laureth sulfate, lauryl sulfate and other ingredients including the silicone premix are added to the finishing tank with ample agitation to insure a homogeneous mixture. A sufficient amount of the silicone premix is added

----- 6513433

classes:1 101/457 1 101/462 1 430/302

score: 349

keywords: semiconductor;titanium oxide;titanium;amide;organic solvent;organic;phthalic;salicylic acid;salicylic;benzoic acid;benzoic;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;ammonium salt;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;glycol;solvent;heated;aluminum;ultrasonic;meta l salt;phenol;hydroxyl;formic;solvents;monoethyl ether;glycol monoethyl;monoethyl;glycol monomethyl;monoethyl;propylene glycol;propylene;triethylene glycol;triethylene;ethylene glycol;amides;amines;corrosion;ethanol;propionic;adipic acid;adipic;lactic;acetic acid;acetic;aliphatic;aryl;alkyl;potassium;sodium salt;urea;anhydride;alkaline;oxides;dissolved;phase;insoluble;alkali;acidic;inorganic;alkanolamine;etching;exposure;semiconductor;titanium oxide;titanium;amide;organic solvent;organic;phthalic;salicylic acid;salicylic;benzoic acid;benzoic;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;ammonium salt ;

----- 6117357

classes:1 252/18638 1 510/313

score: 339

keywords: peroxy;diamine;hydrogen peroxide;peroxide;residue;organic;percarbonate;perborate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;quaternary;peroxide;peracid;perox yhydrate;lactam;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;solvent;disclosures;aluminum;washed;additive;enzyme;sugar;phenol;diethylenetriaminepentaacetic acid;ethylenediaminetetraacetic acid;ethylene diaminetetraacetic;solvents;propylene;amides;ethylenediamine;moiety;acetic;aliphatic;ammonium salts;alkalinity;aryl;alkyl;metal ions;sodium salt;urea;anhydride;alkaline;dissolved;species;alkali;acidic;inorganic;residue;organic;percarbonate;perborate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;quaternary;peroxide;

100% 100% 100% 100%

BALANCE TO:

Note 1: Bleach Activator according to any of Examples I-V.

Note 2: These **hydrogen **peroxide** sources are expressed on a weight % available oxygen basis. To convert to a basis of percentage of the total composition, divide by about 0.15.

Note 3: Transition Metal Bleach Catalyst: Pentaamineacetatocobalt (III) nitrate; may be replaced by MnTACN.

EXAMPLE X

Cleaning compositions having liquid form especially useful for cleaning bathtubs and shower tiles without being harsh on the hands are as follows:

Ingredient	% (wt.)	
	A	B
Bleach Activator*	7.0	5.0
hydrogen **peroxide	10.0	10.0
C.sub.12 AS, acid form, partially neutralized	5.0	5.0
C.sub.12-14 AE.sub.3 S, aci		

----- 6525041

classes:1 514/184 1 514/185 1 540/465

score: 327

keywords: dmso;diamine;hydrogen peroxide;peroxide;residue;dimethyl;amide;organic;hydrogen peroxide;phthalic;benzoic;aromatic;carboxylic acid;carboxylic;peroxide;ammonium;oxide;salt;acid;hydrogen;amine;ether;solvent;dropwise;heated;benzoate;copper;washed;immersed;enzyme;phenol;solvents;triethylene;glycols;amides;amines;diamines;ethanol;acetic;aryl;alkyl;potassium;crystallization;anhydride;alkaline;oxides;dissolved;species;residue;dimethyl;amide;organic;hydrogen peroxide;phthalic;benzoic;aromatic;carboxylic acid;carboxylic;peroxide;

- 51.56, 52.68, 54.18, 62.09, 127.45, 127.66, 129.94, 130.09, 133.96, 134.44, 143.86, 144.46, 168.73; FAB mass spectrum (NBA-Li) m/z 828 [M+H].sup.+.

1.E. Synthesis of

D,L-2,3-Bis(3-hydroxypropyl)-1,4,7,10,13-pentaaazacyclopentadecane

To a stirred suspension of

D,L-5,6-bis(3-hydroxypropyl)-1,10,13-tris(p-toluenesulfonyl)-1,4,7,10,13-pentaaazacyclopentadecane-3,8-dione (5.00 g, 6.08 mmole), prepared as in Example 1D, in anhydrous THF (100 ml) under a dry argon atmosphere was added a solution of 1.0 M LiAlH₄ in THF (76.0 ml, 76.0 mmole) **dropwise** over 5 minutes. The yellow homogeneous solution was refluxed for 30 h (by which time it had become heterogeneous) and was then cooled to 0.degree. C. The mixture was then quenched by the **dropwise** addition of saturated Na₂SO₄ (15 ml) while cooling in an ice bath. The solvent was removed in vacuo and any remaining water was removed by azeotroping with toluene (3.times.500

- xypropyl)-1,4,7,10,13-pentaaazacyclopentadecane prepared as in Example 1E (331 mg, 1.00 mmole) and the solution was refluxed for 2 h and then stirred at room temperature overnight. The solvent was removed in vacuo and the white solid was redissolved in a mixture of THF (20 ml) and ethanol (3 ml) and filtered through Celite.TM. diatomaceous earth. The filtrate was concentrated to a volume of 3 ml, ethanol (3 ml) was added and the solution was **heated** to reflux. THF (20 ml) was added to the solution and the crystals which formed were collected to give 820 mg (69%) of the product as a white solid: FAB mass spectrum (NBA-HCl) m/z (relative intensity) 421/423 [(M-Cl).sup.+, 100/33]; Anal. Calcd. For C₁₆H₃₇N₅ MnCl₂: C, 42.02; H, 8.15; N, 15.31; Cl, 15.50. Found: C, 42.11; H, 8.14; N, 15.29; Cl, 15.59.

Example 2

Stopped-Flow Kinetic Analysis

Stopped-flow kinetic analysis has been utilized to determine whether a

- used to grind the yellow solid potassium superoxide (.about.100 mg). The powder was then ground with a few drops of DMSO and the slurry transferred to a flask containing an additional 25 ml of DMSO. The resultant slurry was stirred for 1/2 h and then filtered. This procedure gave reproducibly .about.2 mM concentrations of superoxide in DMSO. These solutions were transferred to a glovebag under nitrogen in sealed vials prior to loading the syringe under nitrogen. It should be noted that the DMSO/superoxide

solutions are extremely sensitive to water, heat, air, and extraneous metals. A fresh, pure solution has a very slight yellowish tint.

Water for buffer solutions was delivered from an in-house deionized water system to a Barnstead Nanopure Ultrapure Series 550 water system and then double **distilled**, first from alkaline potassium permanganate and then from a dilute EDTA solution. For example, a solution containing 1.0 g of

- potassium permanganate, 2 liters of water and additional sodium **hydroxide** necessary to bring the pH to 9.0 were added to a 2-liter flask fitted with a solvent distillation head. This distillation will oxidize any trace of organic compounds in the water. The final distillation was carried out under nitrogen in a 2.5-liter flask containing 1500 ml of water from the first still and 1.0.times.10.sup.6 M EDTA. This step will remove remaining trace metals from the ultrapure water. To prevent EDTA mist from volatilizing over the reflux arm to the still head, the 40-cm vertical arm was packed with glass beads and wrapped with insulation. This system produces deoxygenated water that can be measured to have a conductivity of less than 2.0 nanomhos/cm.sup.2.

The stopped-flow spectrometer system was designed and manufactured by Kinetic Instruments Inc. (Ann Arbor, Mich.) and was interfaced to a MAC IICX personal computer. The software fo

- with 5 ml of the aqueous buffer solution. The entire injection block, mixer, and spectrometer cell were **immersed** in a thermostatted circulating water bath with a temperature of 21.0+-0.5.degree. C.

Prior to initiating data collection for a superoxide decay, a baseline average was obtained by injecting several shots of the buffer and DMSO solutions into the mixing chamber. These shots were averaged and stored as the baseline. The first shots to be collected during a series of runs were with aqueous solutions that did not contain catalyst. This assures that each series of trials were free of contamination capable of generating first-order superoxide decay profiles. If the decays observed for several shots of the buffer solution were second-order, solutions of manganese(II) complexes could be utilized. In general, the potential SOD catalyst was screened over a wide range of concentrations. Since the initial concentration

----- 6306812

classes:1 510/310 1 8/111 1 8/137 1 134/252 1 134/42 1 252/18633 1 510/220 1 510/221 1 510/224 1 510/302
1 510/303 1 510/304 1 510/311 1 510/312 1 510/313 1 510/314 1 510/372 1 510/376
score: 326

keywords: peroxy;diamine;quaternary ammonium;hydrogen peroxide;peroxide;residue;dimethyl;amide;organic;percarbonate;perborate;persulfate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;quaternary ammonium;quaternary;peroxide;peracid;hydroperoxide;peroxyhydrate;sulfoxide;lactam;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;glycol;solvent;disclosures;distilled;heated;benzoate;aluminum;washed;additive;oxidized;metal salt;enzyme;sucrose;glucose;phenol;hydroxyl;solvents;ethers;propylene;diethylene glycol;diethylene glycol;glycols;amides;amines;corrosion;ethylenediamine;ethanol;monoethanolamine;benzyl;moiety;adipic;acetic;aliphatic;ammonium salts;alkalinity;aryl;alkyl;peracetic acid;metal ions;potassium;ammonia;urea;anhydride;alkaline;oxides;dissolved;phase;species;insoluble;alkali;acidic;decomposition;inorganic;residue;dimethyl;amide;organic;percarbonate;perborate;persulfate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;quaternary ammonium;quaternary;peroxide;

- issolved in warm water before adding to the washing appliance if desired.

EXAMPLE 13

Dithiocyanato Manganese (II) 5,8

Dimethyl-1,5,8,12-tetraazabicyclo[10.3.2]heptadecane Synthesis

##STR55##

Synthesis of 1,5,9,13-Tetraazatetracyclo[11.2.2.2.sup.5,9]heptadecane 1,4,8,12-tetraazacyclopentadecane (4.00 g, 18.7 mmol) is suspended in acetonitrile (30 mL) under nitrogen and to this is added glyoxal (3.00 g, 40% aqueous, 20.7 mmol). The resulting mixture is **heated** at 65.degree. C. for 2 hours. The acetonitrile is removed under reduced pressure. **distilled** water (5 mL) is added and the product is extracted with chloroform (5.times.40 mL). After drying over anhydrous sodium sulfate and filtration, the solvent is removed under reduced pressure. The product is then chromatographed on neutral alumina (15.times.2.5 cm) using chloroform/methanol (97.5:2.5 increasing to 95:5). The solvent is removed under reduced pressure and the resulting

----- 6387927
classes:1 514/311 1 514/314 1 514/367 1 514/374 1 514/375 1 514/395 1 546/174 1 548/159 1 548/180 1 548/2
17 1 548/3051 1 548/3101
score: 326

keywords: dmso;diamine;peroxide;residue;dimethyl;amide;organic;phthalic;salicylic;benzoic;aromatic;carboxylic acid;carboxylic;carbonate;peroxide;lactam;salt;acid;hydrogen;amine;ether;glycol;solvent;dropwise;heated;Tertiary;aluminum;washed;enzyme;sugar;hydroxyl;formic acid;formic;organic solvents;solvents;propylene;glycols;ethanolamine;ethanol;propionic;adipic;lactic;acetic acid;acetic;aryl;alkyl;potassium;crystallization;anhydride;dissolved;phase;insoluble;alkali;acidic;dissolve;inorganic;residue;dimethyl;amide;organic;phthalic;salicylic;benzoic;aromatic;carboxylic acid;carboxylic;carbonate;peroxide;

- ature. Propionylchloride (24.99 g) is then added to the solution and the mixture is allowed to warm to RT over night. After addition of 150 ml of 1N NaOH the layers are separated and the organic solution is **washed** with 1N NaOH and saline. Drying over MgSO₄ and evaporation of solvent gives crude 97, which is recrystallized from AcOEt to give pure 97 as white crystals. M.p. 104-106.degree. C. ESI-MS: 247.1 (M+H).
##STR236##

To a solution of 16.93 g of (i-C₃H₇)₂NH in 100 ml of THF are added **dropwise** 104 ml a 1.6 M solution of n-BuLi in hexane at 0.degree. C. The solution is stirred at 0.degree. C. for 10 min, cooled to -78.degree. C., and a solution of 41.36 g of 97 in 150 ml of THF is added **dropwise** at the same temperature. After 3 additional hours at -78.degree. C. 28.9 g of allyl iodide and 150 ml of THF are added. The mixture is then stirred overnight at -78.degree. C. and poured into when 200 ml of

----- 4102823
classes:1 510/351 1 510/348 1 510/352 1 510/355 1 510/452 1 510/453 1 510/488
score: 321

keywords: organic;perborate;carbonate;oxide;salt;acid;Severe;washed;corrosion;alkyl;potassium salt;potassium;sodium salt;alkaline;dissolved;alkali;inorganic;organic;perborate;carbonate;

- rts, cotton T-shirts and other fabrics such as pillow cases are distributed among various individuals. Each dress shirt and T-shirt is worn for one normal working day under uniform conditions, and the other articles are used for their generally-intended purposes. The soiled clothes and fabrics are then **washed** in an automatic agitating-type washer, for a period of 10 minutes, with detergent solutions at 100.degree. F. The detergents employed are the compositions of Examples II, III and IV at a concentration of 0.9 cup per 23 gallons of water and Tide, a commercially-available built granular detergent marketed by The Procter & Gamble Company, used at varying concentrations. Wash water of 2, 7, and 12 grains/gallon hardness is employed. After washing, the clothes are rinsed (6 spray rinses and one deep rinse) and then dried. Direct visual comparisons are made by a panel of expert graders between pairs of shirts and fabrics worn a

----- 6218351
classes:1 510/311 1 510/376 1 510/500
score: 314

keywords: peroxy;diamine;hydrogen peroxide;peroxide;residue;dimethyl;amide;organic;percarbonate;perborate;persulfate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;quaternary;peroxide;peracid;hydroperoxide;peroxyhydrate;sulfoxide;lactam;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;glycol;solvent;disclosures;distilled;heated;benzoate;aluminum;washed;additive;oxidized;metal salt;enzyme;sucrose;phenol;hydroxyl;solvents;ethers;propylene;diethylene glycol;diethylene;ethylene glycol;glycols;amides;amines;corrosion;ethylenediamine;ethanol;monoethanolamine;benzyl;moiety;adipic;acetic;aliphatic;ammonium salts;alkalinity;aryl;alkyl;peracetic acid;metal ions;potassium;sodium salt;ammonia;urea;anhydride;alkaline;oxides;dissolved;phase;species;insoluble;alkali;acidic;decomposition;inorganic;residue;dimethyl;amide;organic;percarbonate;perborate;persulfate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;quaternary;peroxide;Address;

- queous, 20.7 mmol). The resulting mixture is **heated** at 65.degree. C. for 2 hours. The acetonitrile is removed under reduced pressure. **distilled** water (5 mL) is added and the product is extracted with chloroform

report09996773.txt

(5.times.40 mL). After drying over anhydrous sodium sulfate and filtration, the solvent is removed under reduced pressure. The product is then chromatographed on neutral alumina (15.times.2.5 cm) using chloroform:methanol (97.5:2.5 increasing to 95:5). The solvent is removed under reduced pressure and the resulting oil is dried under vacuum, overnight. Yield: 3.80 g, I (87%).

Synthesis of

1,13-Dimethyl-1,13-diazonia-5,9-diazatetracyclo[11.2.2.2.sup.5.9]heptadecane diiodide

1,5,9,13-tetraazatetracyclo[11.2.2.29.sup.5.9]heptadecane (5.50 g, 23.3 mmol) is dissolved in acetonitrile (180 mL) under nitrogen. Iodomethane (21.75 mL, 349.5 mmol) is added and the reaction is stirred at RT for 10 days. The solution is rotov

----- 6066612

classes:1 510/400 1 510/276 1 510/299 1 510/361 1 510/398 1 510/434 1 510/477 1 510/499 1 510/517 1 510/5

28

score: 312

keywords: peroxy;hydrogen peroxide;peroxide;residue;stripping;dimethyl;percarbonate;perborate;persulfate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;peroxyde;peroxyhydr ate;ammonium;oxide;salt;acid;hydrogen;amine;soluble;glycol;solvent;disclosures;heated;Tertiary;aluminum;a dditive;oxidized;enzyme;hydroxyl;solvents;ethers;propylene;glycols;amides;amines;ethylenediamine;ethanola mine;monoethanolamine;moiety;ammonium salts;alkyl;ammonia;anhydride;alkaline;oxides;phase;species;alkali;inorganic;residue;stripping;dimethyl;percarbonate;perborate;persulfate;hydrogen peroxide;aromatic;carboxylic acid;carboxylic;carboxylate;carbonate;hydroxide;peroxide;

- Sodium C.sub.14 -C.sub.15 alcohol ethoxylate
2.0 2.0 0.0 0.0
(0.5) sulfate

Sodium C.sub.14 -C.sub.15 alcohol ethoxylate (6.5)
0.5 0.5 0.5 1.0
Tallow fatty acid 0.0 0.0 0.0 1.1
Sodium tripolyphosphate
0.0 41.0 0.0 0.0
Zeolite A, hydrate (0.1-10 micron size)
26.3 0.0 21.3 28.0
Sodium **carbonate** 23.9 12.4 25.2 16.1
Sodium silicate (1:6 ratio
2.4 6.4 2.1 2.6
NaO/SiO.sub.2 (46%)
Sodium sulfate 10.5 10.9 8.2 15.0
Sodium perborate 1.0 1.0 5.0 0.0
Poly(ethyleneglycol), MW .about.4000 (50%)
1.7 0.4 1.0 1.1
Citric acid 0.0 0.0 3.0 0.0
Nonyl ester of sodium p-hydroxybenzene-
0.0 0.0 5.9 0.0
sulfonate
Homo-polymeri

----- 6514926

classes:1 510/357 1 510/424 1 510/426 1 510/428
score: 308

keywords: peroxy;diamine;quaternary ammonium;hydrogen peroxide;peroxide;residue;dimethyl;amide;organic;per carbonate;perborate;hydrogen peroxide;aromatic;carboxylate;carbonate;ammonium salt;quaternary ammonium;quaternary;peroxide;hydroperoxide;peroxyhydrate;sulfoxide;lactam;ammonium;oxide;salt;acid;hydrogen;amine;soluble;ether;glycol;solvent;disclosures;distilled;dropwise;heated;benzoate;aluminum;copper;washed;additive;enzyme;sucrose;glucose;phenol;solvents;ethers;propylene;diethylene;ethylene glycol;amides;amines;diamines;benzyl;moiety;aliphatic;ammonium salts;alkalinity;aryl;alkyl;potassium salt;potassium;sodium salt;ammonia;anhydride;alkaline;oxides;dissolved;phase;species;insoluble;alkali;acidic;dissolve;decomposition;inorganic;residue;dimethyl;amide;organic;percarbonate;perborate;hydrogen peroxide;aromatic;carboxylate;carbonate;ammonium salt;quaternary ammonium;quaternary;peroxide;

- of alkyl moiety from 10 + x to 10 + y
NaSKS-6 Crystalline layered silicate of formula .delta.-Na.sub.2
Si.sub.2 O.sub.5

report09996773.txt

NaTS	Sodium toluene sulfonate
NOBS	Nonanoyloxybenzene sulfonate, sodium salt
LOBS	C12 oxybenzenesulfonate, sodium salt
PAA	Polyacrylic Acid (mw = 4500)
PAE	Ethoxylated tetraethylene pentamine
PAEC	Methyl quaternized ethoxylated dihexylene triamine
PB1	Anhydrous sodium **perborate** bleach of nominal formula NaBO ₂ .sub.2.H.sub.2 O.sub.2
PEG	Polyethylene glycol (mw = 4600)
percarbonate	Sodium **percarbonate** of nominal formula 2Na ₂ O ₂ .sub.2 H ₂ O
CO ₂ .sub.3.3H ₂ O	
PG	Propanediol
Photobleach soluble	Sulfonated Zinc Phthalocyanine encapsulated in dextrin polymer
PIE	Ethoxylated polyethyleneimine, water
SRP 3	Sulfonated ethoxylated terephthalate polymer
STPP	Methyl capped ethoxylated terephthalate polymer
Sulfate	Sodium tripolyphosphate, anhydrous
TAED	Tetraacetylethylenediamine
TFA	C16-18 alkyl N-methyl glucamide
Zeolite A	Hydrated Sodium Aluminosilicate, Na ₂ O ₁₂ (Al ₁₀ .sub.2 SiO ₂).sub.12.27H ₂ O; 0.1-10 .mu.m

Zeolite MAP Zeolite (Maximum **aluminum** P) detergent grade (Crosfield)
Typical ingredients often referred to as "minors" can include perfumes,

The following example is illustrative of the present invention, but is not meant to limit or otherwise define its scope. All parts, percentages and ratios used are expressed as percent weight unless otherwise noted.

Example 25
The following laundry detergent compositions A to F are prepared in accordance with the invention.

```
-----      6514944  
classes:1 514/29 1 536/72 1 536/74  
score: 288
```

Keywords: dmso; diamine; hydrogen peroxide; peroxide; residue; dimethyl sulfoxide; dimethyl; organic solvent; organic; hydrogen peroxide; benzoic; aromatic; carboxylic; carbonate; hydroxide; peroxide; sulfoxide; ammonium; oxide; salt; acid; hydrogen; amine; ether; glycol; solvent; distilled; dropwise; heated; Tertiary; aluminum; washed; oxidized; sugar; hydroxyl; formic acid; formic; solvents; ethylene glycol; amines; ethylenediamine; ethanol; moiety; propionic; acetic acid; acetic; alkalinity; aryl; alkyl; potassium; anhydride; dissolved; phase; alkali; inorganic; stripped; residue; dimethyl sulfoxide; dimethyl; organic solvent; organic; hydrogen peroxide; benzoic; aromatic; carboxylic; carbonate; hydroxide; peroxide; Inventors:

- the mixture is stirred for 24 hours at room temperature. The mixture is diluted with ethyl acetate, and this is **washed** with 5% NaHCO₃ solution and brine, dried (Na₂SO₄) and concentrated under reduced pressure. The residue is chromatographed on silica gel (1:1 acetone/hexanes) to give the title compound as a white foam.

Further variations and modifications of the foregoing will be apparent to those skilled in the art and are intended to be encompassed by the claims appended hereto.

Example 29

Fluorination of C2 Position Before Fused Ring Formation

Synthesis of

2'-O-benzoyl-6-O-propargyl-3-descladinosyl-3-oxo-10,11-anhydro-2-fluoro-15-methylerythromycin A

A solution of

2'-O-benzoyl-6-O-propargyl-3-descladinosyl-3-oxo-10,11-anhydro-15-methyl-e rythromycin A in tetrahydrofuran under inert atmosphere is cooled to -78.degree. C. and treated with 1.0 M potassium tert-butoxide in tetrahydrofuran. The mixture is st

```
-----  
6103678  
classes: 1 510/101 1 510/102 1 510/104 1 510/276 1 510/299 1 510/300 1 510/308 1 510/329 1 510/330 1 510/3  
92 1 510/400 1 510/504 1 510/515 1 510/517 1 510/520 1 510/528  
score: 279
```

keywords: peroxy; diamine; quaternary ammonium; peroxide; dimethyl; amide; organic; percarbonate; perborate; persulfate; benzoic acid; benzoic; aromatic; carboxylic; carboxylate; carbonate; quaternary ammonium; quaternary; peroxide; peroxyhydrate; lactam; ammonium; oxide; salt; acid; hydrogen; amine; soluble; ether; glycol; heated; Tertiary; salicylate; aluminum; washed; additive; oxidized; enzyme; hydroxyl; solvents; ethers; diethylene; amides; amines; ethanolamine; ethanol; benzyl; moiety; aliphatic; ammonium salts; alkyl; potassium; anhydride; alkaline; phase; species; alkali; inorganic; dimethyl; amide; organic; percarbonate; perborate; persulfate; benzoic acid; benzoic; aromatic; carboxylic; carboxylate; carbonate; quaternary ammonium; quaternary; peroxide;

- O) spectrum
shows the total shifting of the methylene peaks at 2.5-3.0 ppm to about 3.5 ppm. To the solution is added just enough sodium bisulfite as a 40% water solution to bring the residual **peroxide** level down to 1-5 ppm. The sodium sulfate which forms causes an aqueous phase to separate which contains salts, but little or no organics. The aqueous salt phase is removed and the desired oxidized polyethyleneimine derivative is obtained and stored as a 52% solution in water.

The following are non-limiting examples of the hydrophilic perfume compositions that are used herein:

Perfume ingredients	1 Wt. %	2 Wt. %	3 Wt. %
3,7-Dimethyl-6-octenol	10	--	5
Benzyl **salicylate**	5	20	5
Benzyl acetate	10	15	5
Benzophenone	3		
--			
Nerol	2	--	
Phenoxy ethyl **propionate**	3	--	
Phenylethyl acetate	5	17	
Phenylethyl alcohol	8	17	
alpha-Terpineol	5	13	
alpha-Terpinene	5	--	
Tetrahydromyrcenol	2	--	
Total	100	100	

EXAMPLE 1

The following compositions are in accordance with the present invention

Component	A	B	C	D	E	F	G	H	I	J
DEQA	2.6									
	2.9	18.0								
		19.0								
			19.0							
				19.0						
					--	--	--	--	--	--
TAE25	--	1.0	--	--	--	--	--	--	--	--
Fatty acid	0.3									
	--	1.0	--	--	--	--	--	--	--	--

----- 6510877

classes:1 141/100 1 141/18 1 401/47
score: 275

keywords: peroxy; diamine; hydrogen peroxide; peroxide; amide; organic peracid; organic; percarbonate; perborate; hydrogen peroxide; carbonate; quaternary; peroxide; peracid; hydroperoxide; peroxyhydrate; lactam; ammonium; acid; hydrogen; ether; glycol; solvent; disclosures; aqueous hydrogen; enzyme; glucose; phenol; diethylene glycol; diethylene; ethanol; moiety; alkalinity; alkyl; potassium; urea; alkaline; acidic; inorganic; etching; amide; organic peracid; organic; percarbonate; perborate; hydrogen peroxide; carbonate; quaternary; peroxide;

- y the potassium salts of peroxodisulfuric acid and

mixtures thereof; more preferably, of peroxomonosulfuric acid including the commercial triple-salt form sold as OXONE.TM. by DuPont and also any equivalent commercially available forms such as CUROX.TM. from Akzo or CAROAT.TM. from Degussa. Certain organic **peroxide**'s, such as dibenzoyl **peroxide**, may be useful, especially as additives rather than as a primary active oxygen source. A preferred active oxygen source, as noted, includes peroxyhydrates, sometimes known as peroxyhydrates or peroxyhydrates. These are organic or, more commonly, inorganic salts capable of releasing **hydrogen **peroxide** readily. They include types in which **hydrogen **peroxide** is present as a true crystal hydrate, and types in which **hydrogen **peroxide** is incorporated covalently and is released chemically, for example by hydrolysis. Typically, peroxyhydrates deliver **hydrogen **peroxide** readily enough that it c

- can be extracted in measurable amounts into the ether phase of an ether/water mixture. Peroxyhydrates are characterized in that they fail to give the Riesenfeld reaction, in contrast to certain other active oxygen sources. Peroxyhydrates are the most common examples of "hydrogen **peroxide** source" materials and include the perborates, percarbonates, perphosphates, and persilicates.

Other materials which serve to produce or release **hydrogen **peroxide** are, of course, useful. Mixtures of two or more peroxyhydrates can be used, for example when it is desired to exploit differential solubility. Suitable peroxyhydrates include sodium **carbonate** peroxyhydrate and equivalent commercial "percarbonate" bleaches, and any of the so-called sodium perborate hydrates, the "tetrahydrate" and "monohydrate" being preferred; though sodium pyrophosphate peroxyhydrate can be used. Many such peroxyhydrates are available in processed forms with coatings,

- of cationic peroxyimidic, peroxydicarboxylic or peroxydicarboxylic acids to the wash. An analogous but non-cationic palette of bleach activators is available when **quaternary** derivatives are not desired. In more detail, examples of a cationic bleach activator includes the **quaternary ammonium**-substituted bleach activators of WO 96-06915 to Baillely, et al., published Mar. 7, 1996, U.S. Pat. No. 4,751,015 to Humphreys, et al., issued Jun. 14, 1988 and U.S. Pat. No. 4,397,757 to Bright and Postlethwaite, issued Aug. 9, 1983, EP-B-284292 to Aoyagi, et al., issued Oct. 19, 1994, EP-B-331,229 to Darwent, et al., issued Aug. 18, 1993 and including 2-(N,N,N-trimethyl ammonium) ethyl-4-sulphophenyl **carbonate**-(SPCC); N-octyl,N,N-dimethyl-N 10-carbophenoxy decyl ammonium chloride-(ODC); 3-(N,N,N-trimethyl ammonium) propyl sodium-4-sulphophenyl **carboxylate**; and N,N,N-trimethyl ammonium toluoxyloxy benzene sulfonate. Also useful are cationic

----- 4493773

classes:1 510/331 1 510/329 1 510/350 1 510/470 1 510/500 1 510/504

score: 274

keywords: quaternary ammonium;dimethyl;amide;carbonate;quaternary ammonium;quaternary;ammonium;oxide;acid ;amine;ether;washed;oxidized;glucose;phenol;propylene;amides;corrosion;ethylenediamine;ethanol;diethanolamine;moiety;aliphatic;alkalinity;alkyl;potassium;ammonia;phase;alkali;stripped;dimethyl;amide;carbonate;q uaternary ammonium;quaternary;

- thoxylated alcohol have been removed.

The static control readings were obtained as follows:

A load of clothing was **washed** in a full size washing machine, using the composition given above at a usage concentration of about 1750 parts per million in 171/2 gallons of 95.degree. F. (35.degree. C.) water, having a hardness of about 7 grains per gallon. The composition had a pH of about 8 in the laundry solution. The load consisted of about 33 pieces of clothing and contained cotton, polyester/cotton, nylon and polyester materials, and acrylic. The **washed** load was subsequently placed in an automatic dryer, the drum of which had been cleaned with an alcohol-soaked cloth, and dried for a period of 60 minutes. The fabric load was then removed from the dryer and placed in a grounded Faraday Cage. The overall charge reading of the materials in the Faraday Cage was read and recorded as individual items were removed from the Cage. When all the

----- 6514983

classes:1 514/285 1 514/183 1 514/410 1 546/61 1 546/62 1 546/66 1 548/421

score: 266

keywords: diamine;hydrogen peroxide;peroxide;residue;titanium;dimethyl sulfoxide;dimethyl;amide;organic s

olvent;organic;hydrogen peroxide;aromatic;hydroxide;peroxide;sulfoxide;ammonium;oxide:salt;acid;hydrogen;amine;soluble;ether;solvent;distilled;heated;Tertiary;aluminum;washed;oxidized;enzyme;glucose;solvents;amides;amines;ethanol;benzyl;moiety;lactic;acetic acid;acetic;aliphatic;aryl;alkyl;potassium;alkaline;disolved;phase;insoluble;dioxide;alkali;inorganic;exposure;residue;titanium;dimethyl sulfoxide;dimethyl;amide;organic solvent;organic;hydrogen peroxide;aromatic;hydroxide;peroxide;Inventors;Aoki;Hayashi;

- e times with PBS. A secondary antibody, goat anti-mouse IgG (1 mL) with a biotin tag is added along with 1 mL of a solution containing streptavidin conjugated to alkaline phosphatase and 1 mL of NBT reagent (Vector). The cells are **washed** and changes in gene expression are noted calorimetrically. Four senescence-specific genes--collagen I, collagen III, collagenase, and interferon gamma--in senescent cells treated with the PARP inhibitor of Formula I, II, III, IV, V, VI, VII, or VIII are monitored and the results show a decrease in interferon gamma expression with no observable change in the expression levels of the other three gens, demonstrating that the PARP inhibitors of Formula I, II, III, IV, V, VI, VII, or VIII can alter senescence-specific gene expression.

Example 44

Extending or Increasing Proliferative Capacity and Lifespan of Cells
To demonstrate the effectiveness of the present method for extending the proliferative

----- 5565145
classes:1 510/350 1 510/299 1 510/320 1 510/499 1 510/535
score: 265

keywords: peroxy;organic;percarbonate;perborate;persulfate;aromatic;carboxylic;carboxylate;carbonate;hydroxide;peroxyhydrate;lactam;ammonium;oxide:salt;acid;hydrogen;amine;soluble;ether;glycol;solvent;disclosures;distilled;heated;Tertiary;benzoate;aluminum;washed;enzyme;phenol;solvents;propylene;diethylene;glycols;amides;amines;ethanol;monoethanolamine;benzyl;moiety;acetic;ammonium salts;alkyl;potassium;anhydride;alkaline;oxides;dissolved;phase;species;alkali;dissolve;inorganic;stripped;organic;percarbonate;perborate;persulfate;aromatic;carboxylic;carboxylate;carbonate;hydroxide;

- mate 1.0 -- 1.5 1.0 -- --
Sodium acetate -- 2.5 2.5 -- -- --
Magnesium acetate tetra-
 1.7 -- 1.7 0.1 -- --
hydrate
Magnesium chloride hexa-
 -- 1.7 -- -- 0.1 0.7
hydrate
Sodium **hydroxide**
 5.0 5.0 5.0 5.0 5.0 5.0
Perfume and minors
 Balance to 100%

*(80:20) mixture of tartrate monosuccinate/tartrate disuccinate

EXAMPLE XII

An automatic dishwashing composition is as follows.

Ingredient	% (Wt.)
Trisodium Citrate	15
Sodium **carbonate**	20
Silicate.sup.1	9
Nonionic Surfactant.sup.2	
3	
Sodium Polyacrylate (m.w. 4000).sup.3	
5	
Termamyl Enzyme (60T)	
1.1	
Savinase Enzyme (12T)	